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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

3206. **Review of industrial applications of analysis, control and instrumentation.** *Anal. Chem.*, 1957, **29** (4, Part II). This review includes the following contributions. **Air pollution**, K. Kay (589–604). **Automatic operations in analytical chemistry**, G. D. Patterson, jun. (605–614). **Clinical chemistry**, G. R. Kingsley (615–624). **Coatings**, M. H. Swann, M. L. Adams and G. G. Esposito (624–630). **Essential oils and related products**, E. Guenther, K. Kulka and J. A. Rogers (630–637). **Ferrous metallurgy**, H. F. Beeghly (638–643). **Fertilisers**, J. A. Brabson (643–647). **Food**, H. W. von Loesecke (647–656). **Solid and gaseous fuels**, W. H. Ode (657–669). **Non-ferrous metallurgy**, M. L. Moss (670–678). **Pesticides**, W. E. Westlake (679–683). **Petroleum**, R. L. LeTourneau (684–697). **Pharmaceuticals and related drugs**, R. P. Haycock and W. J. Mader (697–713). **Natural and synthetic rubbers**, M. Tryon (714–721). **Water analysis**, S. K. Love and L. L. Thatcher (722–734).

3207. **The Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy.** American Chemical Society and Spectroscopy Society of Pittsburgh. Pamphlet, March, 1957, 48 pp.—Abstracts of the **131** papers presented covering chemical and spectrographic analysis, X-ray analysis, gas chromatography, flame photometry, nuclear magnetic resonance and KBr pellet technique are published. Some (but not necessarily all) of the papers will appear later in full in various journals.

3208. **The thermal stability of analytical standards. IV.** C. Duval (Lab. de Recherches Microanalytiques, Paris, France). *Anal. Chim. Acta*, 1957, **16** (3), 221–225.—The thermolysis curves and the i.r. absorption characteristics of the following substances have been determined—NaOH, $Na_2S_2O_3.5H_2O$, $SnCl_2.2H_2O$, $AgNO_3$, $CuSO_4.5H_2O$, $Bi(NO_3)_3.5H_2O$, HIO_3, $KClO_3$, NH_4VO_3, Sb K tartrate, oxalic acid, and sulphamic acid.

W. C. JOHNSON

3209. **Evaluation of analytical methods by the sensitivity criterion.** R. D. Stiehler and J. Mandel. *Anal. Chem.*, 1957, **29** (4, Part I), 17A–19A.—The use of the sensitivity criterion for evaluating test methods is discussed and applications are described.

K. A. PROCTOR

3210. **Use of sulphanthrol as a reagent in qualitative analysis.** P. N. Shifrina. *Vestnik Khim. Obsch. im D.I. Mendeleev. Chkalovsk. Otd.*, 1956, (6), 35–37; *Ref. Zhur., Khim.*, 1957, Abstr. No. 4659.—A saturated aq. soln. of sulphanthrol forms white ppt. with Hg^{2+}, Hg_2^{2+}, Ag, Pb, Cd, Sn and Sb; a yellow-brown ppt. with Fe^{3+}; a grey ppt. with Cr^{2+}; and a green ppt. with Cu^{2+}. No ppt. or coloured soln. is obtained with Co, Ni, alkali or alkaline-earth metals. Sulphanthrol completely precipitates Cu^{2+}, and the ppt. obtained is easily filterable. The solubility of this compound is 0·02 g in 100 ml of water at 17°, and it is poor in mineral acids, alkalis, aq. NH_3, alcohol, benzene and CCl_4. The Cu^{2+} comp. contains 9·5% of Cu; the sensitivity of the reaction is 3·17 μg of Cu per ml. No interference is caused by Co, Ni, Mn, Zn, Al, Ag, alkali metals, alkaline earths, small quantities of Sn, Bi or Sb. By using Cu^{2+} it is possible to detect the sulphanthrol in soln.

C. D. KOPKIN

3211. **1:2:5:8-Tetrahydroxyanthraquinone (quinalizarin) as a colorimetric reagent for inorganic cations.** Anil K. Mukherji and Arun K. Dey (Inst. of Chem., Univ. Allahabad, India). *Z. anal. Chem.*, 1957, **155** (6), 417–419 (in English).—The reactions of quinalizarin with 23 metal salts have been studied. The reagent is particularly sensitive for U^{VI}, Fe^{III} and Ca, as well as for Al, B, Be, Ga, Mg, Th, Zr and rare earths.

A. R. ROGERS

3212. **Rhodizonic acid as a reagent in inorganic analysis. I. Colour reactions of sodium rhodizonate with metal ions.** Anil K. Mukherji and Arun K. Dey (Inst. of Chem., Univ. Allahabad, India). *Chim. Anal.*, 1957, **39** (4), 148–149.—The colorimetric reactions of Na rhodizonate with various metal ions have been examined. Freshly prepared aq. soln. of the indicator are brown, but this coloration disappears after one day, owing to decomposition into croconic acid which gives no colour reaction with metal ions. A list of colour changes of the indicator with various metals is given; salts of Ag, Pb and Fe have an especially high sensitivity.

W. J. WRIGHT

3213. **Analytical properties of oxaldihydroxamic acid.** D. Monnier and C. Jegge (Lab. de Chimie Minérale, Geneva Univ., Switzerland). *Helv. Chim. Acta*, 1957, **40** (2), 513–526 (in French).—Three tautomers of oxaldihydroxamic acid (**I**) are possible. From a study of the i.r. spectra of **I** and its sodium salt, two alternatives for the probable structure are suggested. The constants K_1 and K_2 of acid-base combinations, determined potentiometrically by pH measurements, were $1·08 \times 10^{-7}$ and $1·01 \times 10^{-9}$, respectively. The solubility product of the calcium salt of **I**, determined conductimetrically, was $1·8 \times 10^{-8}$, and by pH measurements $1·3 \times 10^{-8}$, at 25°. The formation of at least two complexes of **I** with Fe^{III} in a perchloric medium has been established: an orange complex (Fe to acid, 1:1) and a violet complex (Fe to acid, 2:1), their constants being $3·6 \times 10^{-4}$ and $5·3 \times 10^{-8}$, respectively. The absorption spectra and formulae of the complexes were studied.

W. J. WRIGHT

3214. **Polyanion-stabilised diazonium cations and diazonium spot-test papers as analytical reagents.** J. L. Lambert and V. E. Cates (Kansas State

College, Manhattan, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 508–512.—Diazonium cations are stable and can be stored when ionically bound to nuclear sulphonic ion-exchange resins, or chemically bound to filter-paper via salt formation of an arsonic acid group with Zr. When released, the diazonium ion can, by proper pH adjustment, be made to react with a phenol or an aromatic amine to form an azo dye whose colour and intensity can be used for identification purposes. Colour data at various pH values are listed for $\simeq 30$ hydroxy and amine compounds, for both ion-exchange and test-paper techniques.

G. P. COOK

3215. **Syntheses of derivatives of dithizone and their application to analytical chemistry. I. *pp'*-Dimethyldithizone.** Shinsuke Takei and Kuniko Shibuya (Dept. of Applied Chem., Fac. of Engng, Tohoku Univ., Sakurakoji, Sendai). *Japan Analyst*, 1956, **5** (12), 695–701.—*pp'*-Dimethyldithizone (di-*p*-tolylthiocarbazone) (**I**) was synthesised, and its use for the colorimetric determination of Zn, Cd, Hg and Pb was studied. The solubility of **I** is similar to that of dithizone (**II**), the max. absorption appearing at 456 and 628 mμ (that of **II** at 450 and 620 mμ). The max. absorptions of the Zn, Cd, Hg and Pb complexes of **I** are at 535, 530, 502 and 530 mμ, respectively, at wavelengths slightly longer than those of corresponding **II** complexes. Their extinction coeff. are greater than those of the **II** complexes by $\simeq 20\%$, whilst their formation const. are appreciably smaller.

K. SAITO

3216. **The specificity of Schiff's reaction.** G. Zinner (Inst. f. Pharm. Chemie und Lebensmittelchemie der Univ. Marburg/Lahn). *Z. anal. Chem.*, 1957, **155** (6), 412–415.—It is shown that the pink colour given by N-hydroxypiperidine with Schiff reagent in acid soln. is due to the formation of $C_5H_{11}NO_3S$ (which is probably piperidine-2-sulphonic acid), with regeneration of parafuchsine.

A. R. ROGERS

3217. **The standardisation of permanganate by arsenous oxide.** Rameshwar Prasad and Arun K. Dey (Chem. Lab., Univ. of Allahabad, India). *Z. anal. Chem.*, 1957, **155** (6), 420–423 (in English).—The effect of catalysts, temp. and concn. of acid on the titration of As_2O_3 with $KMnO_4$ is described. The sharpness of the end-point improved with the catalysts in the order Br, KBr, $KBrO_3$, I, KI, KIO_3. With KIO_3, the results are about 0·2% lower than in the presence of the other catalysts. *Procedure*—Dilute the 0·1 *N* As_2O_3 (25 ml) with 4 *N* HCl (10 or 15 ml), add two drops of 0·0025 *M* KIO_3, or of 0·0025 *M* KI, or of a saturated aq. soln. of iodine, and titrate in the cold with 0·1 *N* $KMnO_4$.

A. R. ROGERS

3218. **The constancy of titrations with very dilute ethylenediaminetetra-acetic acid volumetric solutions.** H. Flaschka and F. Sadek (Nat. Res. Centre, Dokki-Cairo, Egypt). *Z. anal. Chem.*, 1957, **156** (1), 23–28.—The influence of glass on 0·001 *M* solutions of EDTA is discussed. The storage of these solutions in plastics bottles is recommended. After 44 days the factor for a 0·001 *M* solution of EDTA stored in a polyethylene container remained at 1·00, whereas for solutions stored in glass bottles it varied from 0·97 to 0·915 according to the treatment of the glass before storage.

M. F. C. LADD

3219. **Complexometric titrations following cupferron separation of interferences.** J. S. Fritz, M. J. Richard and A. S. Bystroff (Inst. for Atomic Research, Iowa State Coll., Ames, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 577–579.—Bivalent metals can be titrated in aq. soln. after extraction (with an organic solvent) of interfering ter- and quadri-valent metals as their cupferron complexes. Data are given for Mg, Ca, Mn, Zn, Cd, Pb, Co and Ni. Several solvents were examined and the most satisfactory was found to be a mixture of equal vol. of benzene and *iso*amyl alcohol. The sample in aq. soln. is mixed with the solvent, the pH is adjusted to between 0·3 and 0·8 with dil. HCl, and cupferron is added. The mixture is shaken, and the aq. layer is separated and titrated with EDTA (disodium salt). Data are given for the removal of Fe, Th, Bi, Zr, Ti and Sn. Separation from Cu, Al or rare earths is incomplete.

G. BURGER

3220. **Potentiometric titrations in non-aqueous solution. I. An empirical acidity-potential scale of twelve solvents.** H. B. van der Heijde and E. A. M. F. Dahmen (N. V. De Bataafsche Petrol. Maatsch., Amsterdam, Holland). *Anal. Chim. Acta*, 1957, **16** (4), 378–391 (in English).—The practical possibility of potentiometrically titrating an acid or base, or mixture of acids or bases, in a non-aq. solvent depends mainly on the "intrinsic" acidity (basicity) of the acid (base) and the limiting potential of the solvent. This possibility, and the selection of the most suitable solvent, can be determined (with sufficient accuracy for analysis) from the relative positions of the half-neutralisation potentials (h.n.p.) of the acids (bases) and the limiting potentials of the available solvents. The limiting acidity potentials of 12 solvents (H_2O, acetone, acetic acid, methanol, pyridine, *iso*propyl alcohol, *n*-butylamine, methyl cyanide, trifluoroacetic acid, chlorobenzene, ethylenediamine, and dimethylformamide) and the h.n.p., in these solvents, of 16 acids and protonated bases are listed. The acids can be titrated with tetra-*n*-alkylammonium bases in pure pyridine and with exclusion of air.

II. Anomalous behaviour of weak oxygen acids in inert and weakly basic solvents. H. B. van der Heijde. *Ibid.*, 1957, **16** (4), 392–400.—The anomalous titration curves of formic, salicylic, acetic, benzoic and toluene-*p*-sulphinic acids, phenol and some derivatives thereof are characterised by reproducible rises of potential in the region of half-neutralisation and by irreproducible potential drops, often at those stages of the titration corresponding to simple fractions of total neutralisation. The effects are ascribed to acid - anion association and to solute-adsorption on the electrode surfaces. The inert solvent used for differential titrations of weak acids should be selected carefully.

W. J. BAKER

3221. **Potentiometric titration of weak acids and bases in dilute aqueous solution.** J. C. Gage (I.C.I. Ltd., Ind. Hygiene Res. Lab., Welwyn, Herts., England). *Analyst*, 1957, **82**, 219–228.—Limitations in the application of the Henderson equation (which is an approximation) to the titration of weak acids and bases are discussed and it is shown that the usefulness of the graphical method of finding the concn. c and the (Brønsted) dissociation constant K may be increased by the use of a more exact form of the equation. The transformation of the titration graph to a linear form is described, and examples are given of the titration with alkali of

chlorophenols at a concn. of 10^{-4} *M*, singly and in admixture, and of quinine and brucine. The potentiometric titration assembly used is described and illustrated. A. O. JONES

3222. **The sensitivity of some chromatographic sprays.** G. B. Heisig and F. H. Pollard (Univ. of Bristol, England). *Anal. Chim. Acta*, 1957, **16** (3), 234–237.—Solutions of 26 different cations, spotted on paper, were sprayed with soln. of 19 different reagents. By using cation soln. of diminishing strength, the most sensitive reagent and the limit of identification of each cation were determined. Observations of both colour and fluorescence reactions were recorded. W. C. JOHNSON

3223. **Studies on paper chromatography. II. Rhythmicity on impregnated filter-papers.** A. C. Chatterji and H. Bhagwan (Dept. of Chem., Lucknow Univ., India). *Z. anal. Chem.*, 1957, **156** (1), 7–15.—Whatman No. 1 paper strips impregnated with soln. of $Pb(NO_3)_2$ and KI were used with and without colloids as fixing agents to study precipitation phenomena. Conditions characterising the formation of uniform bands are described. (*Cf. Anal. Abstr.*, 1956, **3**, 2390.) M. F. C. LADD

3224. **Effect of different carrier gases on retention times in gas-adsorption chromatography.** S. A. Greene and H. E. Roy (Aerojet General Corp., Azusa, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 569–570.—The effect of five different carrier gases (helium, argon, nitrogen, air and acetylene) on the retention times of methane on a 10-ft. activated-charcoal column at 250° has been investigated. The retention times vary from 5 to 34 min. The efficiency of separation of a low-boiling gas mixture depends markedly on the carrier gas (helium or argon) used, as do the retention times of the components. Elution with air, argon or nitrogen also improves the symmetry of the recorded wave forms. K. A. PROCTOR

3225. **Analytical identification by spectrophotometry. The use of ionisation constants of weak acids.** A. I. Biggs (Dept. of Chem., Univ. of Malaya). *Analyst*, 1957, **82**, 274–275.—The u.v. absorption spectrum of a weak acid is often markedly dependent on pH, *i.e.*, in alkaline soln. the absorption of the anion of the acid is being measured and when the pH is reduced (*e.g.*, by addition of 0·1 *N* HCl) the spectrum is that of the uncharged molecule. It is shown that at a more favourable wavelength three measurements of extinction, *viz*, that in a buffered soln. (D), that in an acid soln. (D_1) and that in an alkaline soln. (D_2), suffice to determine the degree of dissociation $\alpha = (D - D_1)/(D_2 - D_1)$. Under standard conditions this may serve to identify the acid. If the concn. of the acid is low, α should be independent both of the concn. and of the presence of impurities, provided that absorption due to impurities does not vary with pH. Such measurements with commercial samples of phenol and nine substituted phenols are recorded and suggest a rapid method of identification particularly useful for impure substances and those having low m.p. or derivatives difficult to prepare. A. O. JONES

3226. **The influence of an inert gas atmosphere on the sensitivity of spectrographic analysis.** E. N. Vinnichenko and A. N. Zaĭdel'. *Vestnik Leningr. Univ.*, 1956, **10**, 22–28; *Ref. Zhur., Khim.*, 1957, Abstr. No. 4688.—The influence of He and A on the sensitivity of the determination of Be, Ga and Cd by arc and spark spectra has been studied. With arc excitation in A, there is a 25-fold strengthening of the Be line 3130·4 A, while the intensity of the other Be lines is increased not more than two to three times. With spark excitation in A there is no strengthening of the lines. The same results are obtained with He. Replacing the air with A does not reduce the intensity of the background. In He the background is reduced, thereby increasing the sensitivity. A study carried out with alloys based on Cd, Al and Mg shows that in an atmosphere of He in a number of cases there is a considerable gain in concn. sensitivity, but the lowering of the background intensity is accompanied by a considerable weakening of the intensity of the lines, which makes necessary a large increase in the exposure time (up to 60 times). It is concluded that replacing the air with A or He is not a general method for increasing the sensitivity of spectrographic analysis, but it is convenient in certain cases. C. D. KOPKIN

3227. **A tracer study on the penetration of a sample solution into the carbon electrode in spectrographic analysis.** Kenjiro Kimura and Kazuo Saito (Dept. of Chem., Univ. of Tokyo, Hongo, Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (1), 1–3 (in English).—The solution arc method of quantitative spectrographic estimation of non-ferrous metals is sensitive but requires a careful procedure in accordance with precise directions. To determine if this drawback was due to differences in the carbon electrodes into which the sample was impregnated, the penetration of a soln. of zinc and lead containing radioactive ^{65}Zn was studied. Impregnated electrodes were sawn at measured intervals and the radioactivity of the dust samples was measured. Penetration by Zn was not uniform and showed a max. at a depth of several millimetres from the top of the electrode. Depth of penetration is independent of apparent hardness and increased with increasing porosity. Preliminary discharge, before impregnation, also increases depth of penetration. Preliminary treatment of the electrode with solvents had little effect. G. BURGER

3228. **Kinetics of the electrolytic deposition of small amounts of metal with simultaneous discharge of hydrogen.** H. Coriou, J. Huré and J. Coursier (Comm. Energie Atomique, Saclay, France). *Anal. Chim. Acta*, 1957, **16** (4), 357–364.—The kinetic influence of temp., c.d., viscosity, concn. of cation and acid, stirring and other factors on the deposition of small amounts of metal on a platinum or mercury electrode during the simultaneous discharge of a large amount of H^+ has been studied by the procedure described previously (Coriou *et al.*, *J. Chim. Phys.*, 1951, **48**, 55). The observed increase in the migration current of the metal ion can be correlated with the effects of the variables. W. J. BAKER

3229. **Use of radioactive isotopes in quantitative analysis.** A. K. Lavrukhina. *Zavod. Lab.*, 1957, **23** (3), 296–303.—A review, with 86 references. G. S. SMITH

3230. **Applications to qualitative and quantitative analysis of the effect of absorption of electrons and X-rays.** S. Yamaguchi (Sci. Res. Inst., Hongo, Tokyo, Japan). *Z. anal. Chem.*, 1957, **156** (2), 109–111.—The use of electron diffraction and X-ray microscopy in analysis is illustrated by the qualitative analysis of Fe_2O_3 - ThO_2 mixtures and

the semi-quantitative determination of Th in crude monazite. A. R. ROGERS

3231. **Chemical determination of different radiations of a nuclear reactor.** J. Pucheault (Inst. Radium, Paris). *J. Chim. Phys.*, 1956, **53** (9), 705–713.—Three soln. are used: (*i*) a soln. of ferric *o*-phenanthroline (ferrine) in 0·1 *N* H_2SO_4 which is reduced by γ-rays to ferrous *o*-phenanthroline (ferroin); the ferroin is determined spectrophotometrically; (*ii*) a soln. of ceric sulphate in 0·8 *N* H_2SO_4 containing boric acid; and (*iii*) a soln. of ceric sulphate in 0·8 *N* H_2SO_4. By comparing the reduction of Ce^{4+} in (*ii*) and (*iii*) the neutronic flux can be calculated. N. E.

2.—INORGANIC ANALYSIS

3232. **The spectrographic analysis of gases in the spark discharge.** A. L. Stolov. *Uch. Zap. Kazansk. Univ.*, 1956, **116** (1), 118–120; *Ref. Zhur., Khim.*, 1957, Abstr. No. 4693.—The analysis of a mixture of N and CO_2 by excitation of their mol. spectra in the high-frequency discharge is described. With this discharge a pressure of 6 cm (Hg) was developed in a quartz water-cooled tube. Besides the band systems noted earlier (Mochalov, *Dokl. Akad. Nauk SSSR*, 1949, **67**, 251; Mizushima *et al.*, *J. Amer. Chem. Soc.*, 1950, **72**, 5176), there are observed bands of the first positive group of N_2, CO_2 bands of the system of Fox, Duffendack and Barker, and CO bands of the first negative and fourth positive systems. The heads of the N_2, CO_2 and CO_2^+ bands served as the analytical pairs of lines. A calibration curve, based on standard mixtures with N content from 1·5 to 33%, was constructed. The error of the analysis is within the limits 1·1 to 2·25%. The lines used analytically are—N_2 3576·9 A, CO_2^+ 2897·5 A; N_2^+ 4278·1 and CO_2^+ 2897·5 A; N_2 3576·9 and CO_2 3370·0 A.
C. D. KOPKIN

3233. **Application of oscillographic polarography to chemical analysis. I. Determination of binary mixtures of lead and thallium and of cadmium and indium.** Mutsuaki Shinagawa, Hideo Imai and Hiroshi Sunahara (Fac. of Sci., Hiroshima Univ., Hiroshima). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1956, **77** (10), 1482–1486.—Multi-sweep oscillographic polarography with a quick response (70 V per sec.) for potential change was applied to the analysis of binary mixtures of metals having similar $E_{\frac{1}{4}}$. Both Pb and Tl are satisfactorily determined on the cathodic branch at −0·43 and −0·48 V *vs.* the S.C.E., respectively. Although In^{III} is subject to a more complicated reaction at the surface of the Hg, the empirical additive law holds for the wave height (< 0·8 m*M*), in its mixture with Cd. The error is ≃2%.

II. Determination of zinc in nickel. Mutsuaki Shinagawa, Hideo Imai and Hiroshi Sunahara. *Ibid.*, 1956, **77** (10), 1486–1489.—A similar method is applicable to a mixture of two ions having similar $E_{\frac{1}{4}}$ but different rates of reduction on the surface of the Hg. The wave of Ni (>1 m*M*) in *N* KCl is negligible compared to that of Zn in an amount equiv. to less than one-tenth that of the Ni. The wave height is proportional to the concn. for < 1·0 m*M* of Zn on both the anodic and the cathodic branch. The determination of Zn (>0·001 m*M*) in Ni can be carried out with an error of ≃2%; the error increases with decrease in concn. of Zn. K. SAITO

3234. **The determination of low concentrations of heavy water by a float method.** J. Spaepen (Study Centre Application Nuclear Energy, Brussels). *Tech.-wetensch. Tijdschr.*, 1956, **25**, 12–14.—The concn. of small quantities ot D_2O in H_2O is determined by measuring the small changes in the density of the sample compared to that of standard H_2O under the same conditions. The difference in density between sample and standard H_2O is measured by the difference in the respective rates of rising or sinking of a carefully calibrated quartz float, which is weighted with Hg and placed in a quartz tube containing either the sample or standard H_2O. The sample tube is kept in a thermostat within ±0·1° and the temp. for each determination is read to ±0·001° with a Beckmann thermometer. The rate of motion of the float is a linear function of temp. if it does not exceed 0·04 mm per sec. The float rate is determined with a chronometer and a cathetometer. Compensations are made for changes in temp., atm. pressure and viscosity. Careful directions are given for the construction of the float and its calibration. Details of the prep. of the sample and standard H_2O are discussed. The value of ρ can be read to $\pm 6 \times 10^{-7}$ g per ml and for samples containing 0·04% of D_2O the uncertainty is ±0·002%. Some other methods give more accurate results, especially at higher concn., but much more complicated apparatus is required.
CHEM. ABSTR.

3235. **Hydrogen counter for analysis of dilute tritium oxide.** J. W. Healey and L. C. Schwendiman (Radiological Sciences Dept., General Electric Co., Richland, Wash., U.S.A.). *Radiation Res.*, 1956, **4**, 278–285.—Hydrogen is generated by reaction of the tritiated water with calcium, and the gas is passed through a counter tube comprising a silvered glass tube (3 in. long × 0·75 in. in diam.) with a tungsten centre wire. Details are given of a suitable quench circuit. Aqueous samples containing as little as 0·01 μC per ml can be analysed with an accuracy of about 4%. G. SKIRROW

3236. **Use of sodium tetraphenylboron for determining potassium in glass.** N. D. Levina and L. I. Panteleeva (Lvov Polytech. Inst.). *Zavod. Lab.*, 1957, **23** (3), 285–287.—The finely powdered sample (0·3 to 0·5 g) is dissolved in HF and the residue after evaporation at 120° to 140° is treated with 20 to 25 ml of hot water, one drop of phenolphthalein soln. and sufficient freshly ignited CaO (≃ 1·5 g giving an excess of 0·3 to 0·5 g) to yield a stable pink colour. The soln. on a water bath is frequently stirred during 1 hr., then filtered, and the residue is washed 10 to 15 times with hot lime water (1·5 g of CaO in 500 ml of water). The filtrate and washings are neutralised with 2 *N* HCl and evaporated to either 10 to 15 ml (with 2 to 5 mg of K present) or 40 to 50 ml (with 15 to 20 mg of K present). After addition of 10% acetic acid to give a pink colour with methyl red indicator, the soln. at 40° to 60° is treated with the calculated amount of a 0·5 to 1% Na tetraphenylboron soln. to give a 5 to 10% excess. After 3 to 5 min. the liquid is placed in ice water for 10 min., the ppt. is collected, washed, dried at 110° to 130°, and weighed. G. S. SMITH

3237. **Micro-analysis with the aid of ion-exchange resins. X. The detection of small amounts of copper with *p*-phenylenediamine and ammonium thiocyanate.** Masatoshi Fujimoto (Chem. Inst., Fac. of Science, Tokyo Univ., Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (1), 87 to 92 (in German).—The reaction of an ammoniacal soln. of a copper salt with *p*-phenylenediamine and NH_4SCN to give a black ppt. affords a highly sensitive test for Cu when carried out on a cation-exchange resin. For max. sensitivity mix a few grains of resin ("1% DVB") on a white spot-plate with one drop of freshly prepared aq. soln. of *p*-phenylenediamine (1%). Allow to stand for about 5 min., add one drop each of sample soln., 2 *N* aq. NH_3 and 0·3% NH_4SCN soln. Set aside for 10 min. and examine the resin with a lens (× 20) for a violet-brown colour. There is interference from Fe^{3+}, Co^{2+}, Ag^+, Cr^{6+} and NO_3^-, and with some ions comparison with a blank is necessary. The limit of detection is 0·02 μg, in a limiting concn. of 1 in 2×10^6.
G. BURGER

3238. **Modifications in the method of determining copper with bis*cyclo*hexanone oxalyldihydrazone.** E. Somers and J. L. Garraway (Long Ashton Res. Sta., Univ. of Bristol, England). *Chem. & Ind.*, 1957, **13**, 395.—Improvement is obtained by more critical control of pH and concn. of NH_4^+ than has previously been thought necessary. To a mineral acid soln. containing ≯ 50 μg of Cu add 10% ammonium citrate soln. (5 ml), then one drop of neutral red indicator. Neutralise with 3 *N* NaOH, then add borate buffer (400 ml of 0·5 *M* H_3BO_3 and 60 ml of 0·5 *M* NaOH) (5 ml) and bis*cyclo*hexanone oxalyldihydrazone [0·5 ml of 0·5% soln. in aq. ethanol (1:1)]. Dilute to 50 ml with water. Measure the colour with a Unicam S.P.600 at 595 mμ, with 4-cm cells. The colour attains maximum intensity within 10 min. and is stable for 3 hr. There is no interference from 100 μg of Fe^{3+}, Ni^{2+}, Co^{2+}, Mn^{2+}, Mg^{2+}, Zn^{2+}, Al^{3+}, CrO_4^{2-} and PO_4^{3-}. The ash of leaves from a wide variety of plants did not significantly affect the determination.
G. S. ROBERTS

3239. **Gravimetric determination of copper, mercury and cadmium with isoniazid.** Tomoyuki Akiyama, Mikisaburo Fujita, Mitsuo Fujiwara and Keimei Kawashima (Kyoto College of Pharmacy, Yamashina, Higashiyama-ku, Kyoto). *Japan Analyst*, 1956, **5** (12), 701–703.—Both Cu^{2+} and Hg^{2+} are quant. pptd. from weakly acid soln. with isoniazid (**I**), and can be separated from Ag, Pb, Zn, Sb, Sn and Fe. The optimum pH value for the pptn. is 4·3 for Cu and 4·8 to 6·0 for Hg. Their mutual separation met with little success even by the use of EDTA or triphosphate. The sample soln. containing ≃ 20 mg of Cu or Hg is treated with 0·1% **I** soln. and the pH adjusted with acetic acid. The ppt. is washed with 0·1% **I** soln. of pH 4·4, then dissolved in dil. HCl, and the metal is re-pptd. as sulphide and determined gravimetrically. The pptn. of Cd with **I** at pH 4·5 (optimum condition) is not quant.
K. SAITO

3240. **Rapid electrolytic method for simultaneous determination of copper and lead in copper alloys.** S. Bertoldi and A. Tartari (L.R.C.A.A. Fiat, Turin, Italy). *Metallurgia Italiana*, 1956, **6**, 272–274.—The alloy is dissolved in the cold in a mixture of HNO_3 and HBF_4 in proportions varying with the nature of the alloy; the greater part of the HNO_3 is removed by gentle heating, the solution is diluted with H_2O, and the electrolysis is carried out in the presence of H_2SO_4. Copper is deposited as metal and the Pb as PbO_2, for which an empirical factor of 0·820, instead of the theoretical, is used for the conversion to Pb. The only interfering element that may be present in the alloys is Mn, which is deposited as MnO_2 together with the PbO_2. In this case the mixed deposit is treated with HNO_3 and H_2O_2, most of the Pb is pptd. as $PbSO_4$, and the Mn is determined volumetrically or photometrically.
L. A. O'NEILL

3241. **Indirect photometric titration of milligram quantities of silver with ethylenedinitrilotetra-acetic acid. A differential null-point method.** S. J. Gedansky and L. Gordon (Dept. of Chem., Syracuse Univ., N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 566–569.—Silver reacts with potassium tetracyanonickelate^{II} in ammoniacal media to release Ni, which is determined by photometric titration. The photometer is adjusted to zero at 440 mμ for the soln. containing potassium tetracyanonickelate^{II} and murexide in aq. NH_3 soln. The sample is added and titrated with EDTA (disodium salt), standardised against Ag by the same procedure. The vol. of titrant added is plotted against extinction, and the end-point is given by the intersection of this plot with the zero line. The soln. being titrated should contain 3 to 10 ml of conc. aq. NH_3 in about 80 ml, giving a pH value between 11·3 and 11·8; large amounts of ammonium salts should be absent. Most metals interfere and must be previously separated. This method is suitable for the range 0·1 to 10 mg of Ag; for larger amounts the end-point can be determined visually.
G. BURGER

3242. **Rapid method for the determination of microgram quantities of silver in litharge and red lead.** E. S. Dicker and E. A. Johnson (Hopkin and Williams Ltd., Freshwater Rd., Chadwell Heath, England). *Analyst*, 1957, **82**, 285–286.—The lead oxide (0·5 g) is dissolved in 1 ml of 5 *N* HNO_3, 1 ml of water and 0·4 ml of 20-vol. H_2O_2, and 5 *N* NaOH soln. is added until the pH is just above 5·1. Drops of HNO_3 are then added until the pH is slightly less than 3·6. To the clear soln., diluted to 50 ml, 0·5 ml of a 0·02% soln. of *p*-dimethylaminobenzylidenerhodanine in abs. ethanol (industrial spirit is less satisfactory) is added and the liquid is mixed by swirling. In the absence of Ag, a clear yellow soln. is obtained; with increasing content of Ag, the colour changes through orange to pink. The colour obtained is compared with that of standards.
A. O. JONES

3243. **Concentration of beryllium in the analysis of beryllium ores.** B. S. Tsȳvina and N. K. Davidovich. *Zavod. Lab.*, 1957, **23** (3), 280–282.—Beryllium is concentrated by co-precipitation with Ti as phosphate in the presence of EDTA. The sample (0·2 to 0·5 g) is treated with HF, the residue after evaporation of the acid is fused with 3 to 4 g of KHF_2, the cooled melt is evaporated with 10 ml of dil. H_2SO_4 (1 + 1) until fuming ceases and then heated at 500° to 600°. The resulting transparent melt is dissolved in 50 to 60 ml of 5% HCl, and the soln. is mixed with 2 ml of a 0·2% soln. of Ti [prepared by dissolving Ti in HCl, or by fusing TiO_2 with $K_2S_2O_7$ and dissolving the melt in dil. HCl (1 + 3)], one to two drops of HNO_3, 10 ml of 15% EDTA soln., and 10 ml of 10% ammonium phosphate soln. The soln. is made just acid to methyl orange with aq. NH_3, boiled for 2 to 3 min. with 10 ml of 15%

Na acetate soln., and set aside for 1 to 1·5 hr. in the cold. The ppt. is collected on a paper containing paper pulp, washed with cold water, and then dissolved in 10 to 20 ml of hot dil. HCl (1 + 4). The filter is washed with hot water, the soln. and washings being collected in the beaker used for the pptn. Hydrogen peroxide (3%) (2·5 ml) is added, the soln. is just decolorised by dropwise addition of 20% NaOH soln. (if the soln. becomes cloudy, one to three drops of NaOH soln. are added to dissolve the ppt.) and transferred to a calibrated flask, where it is treated with the appropriate amount of 20% NaOH soln. The capacity of the flask and the vol. of NaOH soln. are 50 and 1·25 ml for 0·001 to 0·01% of BeO, 100 and 2·5 ml for 0·01 to 0·07% of BeO, and 250 and 6·25 ml for higher concn. of BeO. The soln. is diluted to the mark and, if cloudy, filtered through a dry filter. An aliquot portion containing $<2\mu g$ and $>20\mu g$ is transferred to a 50-ml colorimeter cylinder and treated with 1 ml of 0·01% beryllon II soln. (*cf.* Lukin and Zavarikhina, *Anal. Abstr.*, 1957, **4**, 1447). After 10 to 15 min. the blue - violet colour is compared with that of a scale of standards. Alternatively, a photocolorimeter with a red filter can be used. G. S. SMITH

3244. **Photometric determination of magnesium with Eriochrome black T in cathodic nickel.** H. Pohl (Bundesanstalt für Materialprüfung, Berlin-Dahlem). *Z. anal. Chem.*, 1957, **155** (4), 263–267.—The method is based on the procedures previously described (Noddack *et al.*, *Anal. Abstr.*, 1956, **3**, 1375; Pohl, *Anal. Abstr.*, 1957, **4**, 2901, or in the press), which have been successfully applied to Zn, Al and cast iron. For cathodic nickel it is applicable to contents of Mg of 0·005 to 0·2%. Magnesium in soln. buffered at pH 10·6 forms a water-sol. red complex with Eriochrome black T, which is measured photometrically at 546 mμ. Nickel and accompanying elements are removed by oxine pptn., followed by extraction with oxine - Na diethyldithiocarbamate - $CHCl_3$. The analysis time for three samples and a blank is approx. 5 hr. The range of error is ±10% (based on five samples). D. F. PHILLIPS

3245. **Calcein as an indicator for the titration of calcium with ethylenediaminetetra-acetic acid.** B. M. Tucker (C.S.I.R.O. Division of Soils, Adelaide, S. Australia). *Analyst*, 1957, **82**, 284–285.—Calcein (fluorescein complexone) is more sensitive than murexide as an indicator for Ca^{2+}. The colour change on titrating calcium soln. with EDTA at above pH 12 is from a bright green fluorescence to a pinkish orange, and the end-point is improved by addition of thymolphthalein, with a sharp change from green to purple at the end-point. For titration of Mg, the end-point is not so satisfactory. For the titration of Ca in the presence of Mg, the Mg must be completely pptd. as $Mg(OH)_2$ by addition of sufficient alkali. If a large proportion of Mg is present, sucrose and Na_2CO_3 and a drop of Nile blue A soln. are added to the neutral sample soln., 10% NaOH soln. is added until the pink colour of the dye is reached, then 2 to 2·5 ml more of the alkali soln. is added and, after addition of the indicator, the liquid is titrated with EDTA. A. O. JONES

3246. **Rapid determination of lime in blast-furnace and open-hearth-furnace slags by the EDTA titration.** Shizo Hirano and Moriji Kurobe (Inst. Techno-anal. Chem., Fac. of Engng, Nagoya Univ., Chigusa-ku, Nagoya). *Japan Analyst*, 1956, **5** (12), 680–684.—The EDTA titration of Ca (10 to 20 mg) is markedly affected by the presence of Al, Fe and Mn (>2 mg each), but this effect is eliminated by the addition of triethanolamine (**I**); Al_2O_3 (<42%), P_2O_5 (<10%), MnO (<6·8%), Fe_2O_3 (<30%) and MgO (<68%) are thus masked for the titration of CaO (≃40%) in the slag. In the presence of a large amount of Mn, an HNO_3 soln. of the sample is treated with $KClO_3$ to precipitate MnO_2. The time taken for an estimation is ≃ 10 min. and the error is <0·5%. *Procedure*—The sample (0·3 g) is treated with $HClO_4$ (15 ml) and made up with water to 200 ml. A 25-ml portion is partly neutralised with 3 *N* NaOH (3 ml), then mixed with **I** (3 ml) and *N* NaOH (3 ml) and titrated with 0·1 *N* EDTA (disodium salt), with murexide as indicator. The time taken for the decomposition of the sample is appreciably reduced by the addition of HF. K. SAITO

3247. **The application of flame photometry to calcium, strontium, barium and lithium.** T. Hollander, A. J. Borgers and C. T. J. Alkemade (Rijksuniv., Utrecht, Netherlands). *Appl. Sci. Res.*, 1956, **58**, 409–427 (in English).—Test soln. of these metals were sprayed into a flame photometer of simple design. With each element, readings were made with a series of interference filters of varying central wavelength with an acetylene - air and propane - air flame for comparison. Conclusions can be drawn on the optimum measuring conditions to be used in routine analyses. An approach is made to the quant. understanding of the form of the standard graphs with Ca, Sr and Ba in the lower concn. range based on the underlying ionisation and dissociation processes. A characteristic saturation effect of the photomultiplier tube used is reported. CHEM. ABSTR.

3248. **Investigation of chemical separation methods for calcium and strontium. Quantitative determination of both elements in calcium - strontium mixtures by the isotopic dilution method.** G. Leliaert and J. Eeckhaut (The Univ., Ghent, Belgium). *Anal. Chim. Acta*, 1957, **16** (4), 311–320 (in English).—Analyses with radiochemical tracers show there is no satisfactory quant. separation of Ca from Sr in mixtures of their nitrates; accuracy is attainable in some instances (extraction with HNO_3 or an ethanol - diethyl ether mixture) only by compensation of errors. Separation by selective pptn. is seldom satisfactory, although very pure $Sr(NO_3)_2$ can be obtained by double pptn. with conc. HNO_3. A semi-micro isotopic dilution method of determining ≃ 20 mg of Ca and Sr (in approx. equiv. amounts) in mixtures of their salts is described. *Procedure*—To the neutral soln. of the sample add known amounts of ^{89}Sr and ^{45}Ca, followed by dropwise addition (with stirring, 30 to 40 min.) of HNO_3 (d = 1·51) until the final d is 1·45. Filter off the ppt. of $Sr(NO_3)_2$, redissolve it, and purify by a double pptn.; dry at 135° and weigh. Evaporate the filtrate to dryness, digest the residue with 10 ml of HNO_3 (d = 1·468) for 15 min. and filter. Evaporate the filtrate to dryness with an excess of H_2SO_4 and weigh the ppt. as $CaSO_4$. Calculate the specific activities (and thence the percentage of Sr and Ca) from the activities of a known wt. of each pure fraction and of the tracer added. The error is ≃ + 3% for both Sr and Ca. W. J. BAKER

3249. **Spectrophotometric determination of zinc and other metals with $\alpha\beta\gamma\delta$-tetraphenylporphine.** C. V. Banks and R. E. Bisque (Inst. for Atomic Res., Iowa State Coll., Ames, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 522–526.—The Zn complex with $\alpha\beta\gamma\delta$-tetraphenylporphine in glacial acetic acid has an absorption max. at 551 mμ which can be used to determine trace amounts of Zn in other metals. With samples that are soluble in glacial acetic acid, namely the alkali metals, magnesium and most of the rare earths, the reagent (*cf.* Priesthoff and Banks, *J. Amer. Chem. Soc.*, 1954, **76**, 937) is added direct to the acid soln. Samples of beryllium, iron and yttrium are dissolved in hot dil. formic acid, the resulting soln. is evaporated to dryness, and the residue is dissolved in acetic acid before addition of the reagent. Samples of cadmium are dissolved in dil. HNO_3, evaporated, and ignited at 500° to 600° before being dissolved in hot glacial acetic acid. In each instance, the extinction is measured 1 hr. after the addition of the reagent. Calibration curves are prepared, and interferences are corrected for by means of appropriate blanks. A method for the indirect determination of some other metals is also described.

G. BURGER

3250. **Determination of small amounts of zinc in ores and in metals with a high content of iron.** N. P. Strel'nikova (Norilsk Mining Metallurgy Combine). *Zavod. Lab.*, 1957, **23** (3), 277–278.—The sample (1 g) is attacked with HCl, then HNO_3, and evaporated to fuming with H_2SO_4. After dilution, the soln. is filtered and made up to 200 ml. An aliquot portion (2 to 5 ml) is mixed with three times its vol. of conc. HCl in a separating funnel, and shaken energetically for 10 to 15 sec. with 3 ml of amyl acetate. The aq. layer is removed and re-extracted with further 3-ml portions of amyl acetate until colourless. Traces of Zn are removed from the combined extracts by shaking with 5 to 6 ml of dil. HCl (1 + 1), and the combined soln. containing the Zn are made just acid to methyl orange indicator by means of aq. NH_3 and dil. H_2SO_4 (1 + 1), then diluted to 25 ml with water, treated with 10 ml of NH_4SCN soln. and two to three drops of dil. H_2SO_4 (1 + 1), and shaken with 25 ml of *iso*amyl alcohol in a separating funnel. The alcoholic layer is separated and washed with 20 ml of a soln. of NH_4SCN in dil. H_2SO_4. The washing is repeated three times. The Zn is extracted and separated from traces of Fe by shaking the alcoholic soln. with two 10-ml amounts of an ammoniacal soln. (composition not stated). The ammoniacal extracts are carefully neutralised to Congo red indicator with dil. H_2SO_4 (1 + 4), avoiding excess of acid, then treated with ten drops of 2% hydroxylamine soln. followed by Na acetate soln. added dropwise until the colour changes from blue to red and then 20 drops in excess; 20 drops of 50% $Na_2S_2O_3$ soln. and 10 ml of 0·005% dithizone soln. are then added and the mixture is energetically shaken for 2 min. The extract containing zinc dithizonate is poured into a colorimeter cylinder and mixed with three drops of Na acetate soln. A blank is carried out simultaneously and mixed with portions of a standard soln. of Zn until the colour matches that of the sample soln. The method is suitable for the determination of 0·002 to 0·01% of Zn in samples containing 50% of Fe. Copper does not interfere; $Na_2S_2O_3$ prevents the formation of a dithizone complex.

G. S. SMITH

3251. **Amperometric determination of microgram amounts of zinc and copper.** A. P. Vol'loshnikova, M. T. Kozlovskiĭ and O. A. Songina (Acad. Sci. Kazakh SSR). *Zavod. Lab.*, 1957, **23** (3), 273–276.—In the amperometric determination of Zn in acid soln. with $K_4Fe(CN)_6$ soln., the solubility of the ppt. must be repressed. With large amounts of Zn the addition of $(NH_4)_2SO_4$ to give a 0·5 *M* soln. is suitable, but with very small amounts, *e.g.*, 6 μg in 20 ml, addition of ethanol (50% of the vol. of the soln.) is necessary to give results of the highest accuracy, but in this case no ppt. is formed. Ethanol cannot be used in the presence of various other ions which would give ppt. Small amounts of Fe do not interfere if the titration is carried out in ammoniacal soln.; zinc adsorbed on the ppt. of $Fe(OH)_3$ is set free during the titration. Large amounts of Fe do not interfere if 2 g of citric acid is added for each 20 ml of soln., which is then neutralised with aq. NH_3 to a bluish-violet colour with crystal violet indicator. Small amounts of Cu can be determined amperometrically by the iodide method. To prevent interference from Fe, the soln. in H_2SO_4 is treated with 3 *M* Na acetate until a red colour appears. After decolorisation with 10 *M* NH_4F and addition of two to three drops in excess, the soln. (20 ml) is carefully neutralised with 4 *N* H_2SO_4 in the presence of crystal violet indicator to the transition from violet to blue. The soln. is titrated amperometrically with a rotating platinum electrode with $Na_2S_2O_3$ soln. after addition of KI. The method is suitable for determining 6 to 60 μg of Cu in the presence of 6000 times as much Fe.

G. S. SMITH

3252. **Chronopotentiometric analysis in fused lithium chloride - potassium chloride. [Determination of cadmium.]** W. S. Ferguson (Univ. of Illinois, Urbana, U.S.A.). *Dissert. Abstr.*, 1956, **16** (9), 1593.—Cadmium, in concn. from 0·7 to $26·6 \times 10^{-4}$ *M* $CdCl_2$, can be determined chronopotentiometrically in a fused LiCl - KCl (eutectic) solvent at 450° with micro-electrodes of platinum wire (2·7 mm × 1·024 mm) or foil (2·79 mm × 3·13 mm). Transition times (0·7 to 5 sec.) can be measured to within ±2%. The diffusion coeff. of Cd^{2+} at 450° in the eutectic solvent is $1·96 \times 10^{-5}$ sq. cm per sec. Suitable cells for the analysis, a robust non-polarisable platinum foil - Pt^{II} reference electrode, and the prep. of fused salt that prevents hydrolytic decomposition during fusion are described.

W. J. BAKER

3253. **The estimation of sub-microgram quantities of mercury.** F. J. Roth (State Dept. Agric., Bur. Chem., Sacramento, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 302–305.—The exact conditions of digestion of the sample in Klein's procedure (*Brit. Abstr. C*, 1953, 243) have been modified. The sample (2 to 10 g) is heated, under a 60-cm 10-bulb Allihn condenser, in a 500-ml flask with H_2SO_4 and HNO_3, a jacketed 15-watt light bulb being used as a source of heat. A visual end-point is used for the final titration with dithizone. For 1 μg of Hg added to garlic, the average recovery was 105%.

A. A. ELDRIDGE

3254. **Direct titration of mercury(I) with potassium permanganate.** I. M. Issa, H. Khalifa and M. Hamdy (Univ. Cairo, Giza, Egypt). *Anal. Chim. Acta*, 1957, **16** (4), 301–304.—Solutions containing Hg^+ can be titrated accurately with $KMnO_4$ in the presence of 0·5 to 1 *N* H_2SO_4 and 1 to 1·5% aq. NaF soln. at 35° to 50°. Sharp end-points are

obtained when the max. final concn. of Hg^+ in the mixed reagents is 0·005 *N*; at higher concn. the titration is unsatisfactory. The soln. of NaF should always be added after the H_2SO_4. The results are within ±0·5% of those obtained by the chloride method. W. J. BAKER

3255. **Micro-volumetric determination of mercury with thionalide.** C. Cimerman and S. Frenkel (Israel Inst. Technol., Haifa, Israel). *Anal. Chim. Acta*, 1957, **16** (4), 305–311 (in English).—The method described depends on the quant. pptn. of $(C_{10}H_7NHCO{\cdot}CH_2S)_2Hg$ when thionalide reacts with Hg^{2+} in acid soln. *Procedure*—To the sample soln. (containing ≯ 4 mg of Hg^{2+} in 0·5 to 1·5 ml) add 1·5 ml of 2 *N* HNO_3 and make up the vol. to ≃ 8 ml. Heat the soln. to ≃90° and add dropwise, with stirring, a two to three-fold excess (≃ 8 mg per mg of Hg^{2+}) of a freshly prepared soln. of thionalide in glacial acetic acid. When the soln. has cleared, filter through a preheated glass filterstick (medium porosity) and wash the ppt. and apparatus until the washings are free from thionalide. Dissolve the ppt. in 4 to 5 ml of a warm mixture (5 + 1) of glacial acetic acid and 5 *N* HCl. Transfer the soln. to a dry flask and, while bubbling CO_2 through it, add KI (20 mg) and an excess of 0·01 *N* KIO_3 - KI soln. Dilute to ≃ 40 ml and back-titrate the soln. with 0·01 *N* $Na_2S_2O_3$ (starch indicator). Deduct from the reading the blank for the reagents. The max. error is ±0·5%; one determination takes ≃ 1 hr. W. J. BAKER

3256. **Determination of mercury in presence of halides. Rapid amperometric titration.** E. P. Parry (Union Oil Research Center, Brea, Calif.). *Anal. Chem.*, 1957, **29** (4, Part I), 546–549.—An amperometric titration of the iodomercurate^{II} ion with di(ethylenediamine)copper^{II} ion is described for the determination of Hg^{II} in the presence of halides. To the sample, containing Hg^{II}, add *M* KI until the ppt. formed redissolves, add conc. aq. NH_3 to pH 9 (thymol blue) and dilute to a known volume. To an aliquot add 2 *M* KNO_3 (7·5 ml), *M* KI (2·5 ml), 0·1% gelatin soln. (5 ml) and H_2O to 50 ml. The concn. of Hg should be 0·02 to 0·06 *M*. Add Na_2SO_3 (1·25 g) to remove oxygen. Place the dropping mercury electrode and agar bridge (satd. KCl) to a depth of ⅛ in. into the soln. and stir for 5 min. Titrate with 0·5 *M* di(ethylenediamine) - $Cu(NO_3)_2$, previously standardised against HgO, to obtain readings in the range 17 to 36 μamp. at −1·1 V (*vs.* the S.C.E.). The average deviation is 0·15% with 0·02 to 0·06 *M* Hg. A const. error of 0·5% is avoided by the standardisation. Cations that react with iodide or are reduced at potentials more positive than −1·1 V interfere. Cyanide must be removed. G. BURGER

3257. **Determination of a small amount of mercury in the presence of a large amount of chloride and the equilibrium of "enolised" dithizone - mercury compound.** Takio Kato, Shinsuke Takei and Akio Okagami (Dept. of Applied Chem., Fac. of Engng, Tohoku Univ., Sakurakoji, Sendai). *Japan Analyst*, 1956, **5** (12), 689–695.—The determination of Hg (< 25 μg) in the electrolytic soln. for the mercury process was studied by the use of EDTA and 1 : 2-diamino*cyclo*hexane-NNN′N′-tetra-acetic acid (**I**) as masking agent for Cu, Zn, Pb and Cd. The extraction of Hg with dithizone in CCl_4 is retarded by the presence of EDTA (≃ 0·01 *M*), but not by that of **I**. The masking of Cu with EDTA (> 1 mg) is not quantitative at pH <4. The reduction of ClO^- or Cl is best achieved by the addition of $NaHSO_3$. *Procedure*—Adjust the pH of the sample soln. (1 to 25 μg of Hg) to 5 with an acetate buffer and add EDTA or **I** and a small amount of $NaHSO_3$. Shake the product with dithizone in CCl_4 (2×10^{-5} *M*) for 2 min. Filter the CCl_4 layer through a dry paper, and measure the extinction at 490 mμ. K. SAITO

3258. **Chelatometry. III. The volumetric determination of aluminium with ethylenediaminetetra-acetic acid (EDTA).** W. Johannsen, E. Bobowski and P. Wehber (Lab. der Metallhütte Mark A.-G., Hamburg-Wilhelmsburg, Germany). *Metall*, 1956, **10** (5–6), 211–212.—A method is described for the determination of Al in its salts and in alloys such as ferrotitanium. *Procedure*—The pH is adjusted to between 1 and 2 with HCl and excess of EDTA (disodium salt) soln. is added. The soln. is buffered to pH 5·5 with solid Na acetate, and NH_4SCN (2 g) and Variamine blue indicator are added. The soln. is titrated to a violet end-point with a mixture of $ZnCl_2$ (0·1 *M*) and $CuCl_2$ (0·005 *M*). The accuracy of the method is greater than ±0·5%. For ferrotitanium the powdered alloy (1 g) is dissolved in a mixture of 6 *N* HCl (50 ml) and HNO_3 (5 ml). The soln. is diluted to 500 ml, solid NaOH is added in excess, and the soln. is then heated for 3 min. and filtered. The filtrate is made up to 500 ml and a 100-ml portion is acidified. Aq. oxalic acid (1% soln.; five drops) is added and the titration carried out as above. The accuracy of the method is better than ±0·1%. C. A. SLATER

3259. **Improved succinate method for the determination of aluminium in ferrous materials.** R. I. Parker (Dept. of Metallurgy, Tech. Coll., Wednesbury, Staffs.). *Metallurgia, Manchr*, 1957, **55**, 103–106.—The method described is suitable for samples containing a large proportion of Fe. The solution (in HCl) of the sample is reduced by boiling with $K_2S_2O_5$, adding (after 1 to 2 min.) succinic acid, urea and quinol; $NaHCO_3$ is then added to give a permanent ppt., which is redissolved in the min. amount of 15% HCl. The solution is diluted, boiled until pptn. is complete, and filtered, and the filter and ppt. are ignited and weighed as Al_2O_3. Titanium interferes and, if present, must be determined and deducted (as TiO_2) from the residue wt. The method is unsuitable for small amounts of Al, *e.g.*, in aluminium-killed mild steel, and in samples containing F. S.C.I. ABSTR.

3260. **Separation of aluminium from phosphorus by means of Trilon B [EDTA].** V. A. Akhvonen (Inst. of Geol. Sci., Acad. Sci., U.S.S.R.). *Zavod. Lab.*, 1957, **23** (3), 295.—In the presence of EDTA, Al is quant. separated from PO_4^{3-} by pptn. with cupferron at pH 3 to 4·6 (bromophenol blue indicator). G. S. SMITH

3261. **A method for the qualitative analysis of mixtures of aluminium oxide and thorium oxide with the aid of X-radiography and electron diffraction.** Shigeto Yamaguchi (Sci. Res. Inst. Ltd., 31, Kamifuji, Hongo, Tokyo, Japan). *Z. anal. Chem.*, 1957, **156** (1), 16–17.—X-ray and electron diffraction photographs of a 1 : 10 mixture of Al_2O_3 and ThO_2 are given. M. F. C. LADD

3262. **Flame-spectrophotometric determination of copper, nickel and manganese in aluminium-base alloys.** J. A. Dean and C. Cain, jun. (Univ. of Tennessee, Knoxville, U.S.A.). *Anal. Chem.*, 1957,

29 (4, Part I), 530–532.—The diethyldithiocarbamates of Cu, Ni and Mn are extracted with $CHCl_3$ from buffered aq. soln. All interferences except that from Co are eliminated and the $CHCl_3$ increases the emission. Dissolve the alloy, evaporate the soln. to dryness, dissolve the residue in HCl, filter, if necessary, and dilute with H_2O. To an aliquot containing 25 to 500 μg of the least abundant metal add 10% Na citrate soln. ($\simeq$ 1 ml for each 10 mg of sample). Adjust to a pH of 6·0 to 6·5 with acetate buffer soln. (pH 6·75), add an excess of 10% Na diethyldithiocarbamate soln. and extract with $CHCl_3$. Aspirate the $CHCl_3$ soln. into the oxy-acetylene flame and read the line and background emission at 3247 and 3253, 4032 and 4010, and 3525 and 3535 A for Cu, Mn and Ni, respectively. Bracket the unknown samples with standards and read the amount of metal present from the appropriate calibration curve. If the content of Cu is more than 20 times that of Ni or Mn, remove 90% with thioacetamide and estimate Cu on a separate sample. Analyses of NBS alloys gave coeff. of variation for replicates (varying from 3 to 9 in number) which ranged from 1·2% to 3·3% for Cu, 0·7% to 1·6% for Ni, and 3·0% to 5·0% for Mn.

G. BURGER

3263. **Detection of thallium with methyl violet.** M. Kovařík and M. Moučka (Montan-Hochschule, Ostrava, Czechoslovakia). *Anal. Chim. Acta*, 1957, **16** (3), 249.—To one drop of the test soln. in a micro test-tube add one drop of bromine water, a few crystals of sulphosalicylic acid, a few drops of conc. H_2SO_4 and two drops of a 0·2% aq. soln. of methyl violet, and shake with 0·2 ml of benzene. If Tl is present the benzene is coloured blue. A positive reaction is given also by Hg, Au, Sb and MoO_4^{2-}, but the first three can be removed by deposition on copper. The blue colours given by VO_3^- and WO_4^{2-} are not extracted by benzene.

W. C. JOHNSON

3264. **The gravimetric determination of thallium(I) as thallium(I) tetraphenylboron.** W. W. Wendlandt (Texas Technol. Coll., Lubbock, U.S.A.). *Anal. Chim. Acta*, 1957, **16** (3), 216–220.—Thallous ions are pptd. quant. by $[B(C_6H_5)_4]^-$ when the acid concn. of the soln. is $< N$. The ppt., $Tl[B(C_6H_5)_4]$, is stable at temp. up to 180°. *Procedure*—To prepare the reagent soln. dissolve 2 g of Na tetraphenylboron in 100 ml of water, add 0·5 ml of M $Al(NO_3)_3$ and 0·5 ml of M NaOH. To an aliquot of the test soln. containing 0·017 to 0·042 g of Tl add 40 ml of water, 20 ml of 2 M Na acetate, two drops of 0·1% aerosol soln. and a slight excess of the reagent soln. Set aside for 2 hr. and filter in a porcelain crucible; wash with water (3 × 10 ml), dry at 100° to 105° for 4 hr. and weigh. Of 33 other cations tested, a ppt. was obtained in the presence of K, Rb, Cs, Zn, Hg^+, Hg^{2+}, Ag, Ce^{4+}, Zr^{4+}, Co, Ni, Mn^{2+}, Pt^{4+}, Sb^{3+}, Bi^{3+} and NH_4^+, but in certain of these ppt. no metal was found.

W. C. JOHNSON

3265. **Determination of low contents of thallium in ores.** I. A. Blyum and I. A. Ul'yanova (Central Lab. of the "Uraltsvetmetrazvedka" Trust). *Zavod. Lab.*, 1957, **23** (3), 283–284.—The sample (1 g) is treated with aqua regia, followed by two evaporations to dryness with HCl, and the residue is taken up in 20 ml of dil. HCl (2·5%). The hot soln. (or the cooled soln. if much Pb is present) is filtered and the insol. matter is washed with water. The filtrate (30 to 35 ml) is boiled and a copper wire (diameter 2 to 3 mm), previously cleaned with HNO_3 and water and bent into a spiral, is immersed therein for 2 to 3 min. The wire is removed, washed and examined for stains. If staining has occurred the wire is cleaned and re-immersed in the boiling soln. The operation is repeated until no further staining occurs and the wire is then left in the boiling soln. for a further 5 min. By this means 10 mg of Sb, 10 mg of Hg and 1 mg of Au can be removed from the soln. in 15 min. The liquid is cooled (a ppt. of cuprous salts may form) and, if the content of Fe is low, 50 mg of Fe^{3+} as $FeCl_3$ soln. is added to serve as a catalyst. The soln. (20 to 25 ml) is cooled and the Tl^+ are oxidised by dropwise addition during stirring of H_2O_2 (30%) so that the cuprous salts are dissolved and an excess of five to six drops is added. After 30 to 40 min. the soln. diluted to 40 ml in a separating funnel is treated with ten drops of 0·2% methyl violet soln. and the coloured compound of Tl^{III} is extracted with 10 ml of toluene. If the amount of Tl exceeds 7 to 10 μg, the extraction is repeated. The extinction of the extract, diluted if necessary to 20 ml, is measured on a photometer with a green filter, or the colour intensity is compared visually with a scale of standards. The method is applicable to practically all ores with a content of Tl of 0·0002 to 0·0025%, and the error of a determination of 10 μg of Tl is $\pm$10%.

G. S. SMITH

3266. **The distribution of metal - 8-hydroxyquinoline compounds between water and organic solvents. II. Studies on 8-hydroxyquinoline and scandium hydroxyquinolinate in the water - benzene system.** F. Umland and H. Puchelt (Inst. für Anorg. Chem., Tech. Hochschule, Hannover). *Anal. Chim. Acta*, 1957, **16** (4), 334–345.—A method is presented for the photometric determination of small quantities of Sc in minerals as the 8-hydroxyquinolinate after extraction from aq. soln. with benzene. The spectra in benzene and the distribution between benzene and water of 8-hydroxyquinoline and Sc 8-hydroxyquinolinate were studied. Scandium 8-hydroxyquinolinate showed an absorption max. at 378 mμ; free 8-hydroxyquinoline showed no measurable absorption at this wavelength. The reagent and its scandium salt were both extracted quantitatively with benzene at a pH of 10·2 $\pm$ 0·2. Hydrolysis of Sc 8-hydroxyquinolinate occurred unless at least 20 mol. of 8-hydroxyquinoline per atom of Sc were present. The method proved suitable for the determination of 3 to 250 μg of Sc. Only phosphate and EDTA interfere. The mean error in the range 40 to 225 μg of Sc was $\pm$2·1%, which is better than that of previously known methods.

G. BURGER

3267. **Separation of quadrivalent cerium from tervalent lanthanons by means of cation exchange.** Yoshio Okabe (Electrotech. Lab., Tokyo). *Bull. Electrotech. Lab., Tokyo*, 1955, **19**, 801–805.—Quadrivalent Ce was separated from weakly acid soln. of tervalent lanthanons by means of cation-exchange adsorption. The distribution coeff. of ceric basic nitrate greatly decreased as the H^+ concn. in Amberlite IR-120 was lowered when a batch method was adopted. The effects of pH, H^+ concn. in the resin and flow rate were examined when impure ceric soln. was passed through a column consisting of $\simeq$ 30 ml of 50 to 70-mesh H - NH_4-type Amberlite IR-120. The efficiency of separation became greater when the pH of the soln. increased and the H^+ concn. in the resin decreased. The effect of flow rate was slight in the range of 1·5 to 4·0 cm per min. At the best conditions,

about 99% pure ceric soln. was recovered with 91% yield from 30% ceric material soln. when a nitrate soln. of pH 1·50 was passed through a column of 5 molar percentage H - NH_4-type resin.
CHEM. ABSTR.

3268. **Volumetric determination of tin in copper alloys.** F. K. Bischitzky. *Metall*, 1956, **10** (21–22), 1041–1042.—*Procedure*—Alloy turnings (1 to 2 g) are dissolved in Cl-free HNO_3 (35 to 50 ml) with cooling. The soln. is set aside at room temp. for 2 hr. and aq. H_2SO_4 (1:1 by vol.) (20 ml) is added. After boiling until SO_2 is evolved, aq. HCl (1:1 by vol.) (25 ml) is added, followed by reduced iron powder. The soln. is filtered and to the filtrate is added conc. HCl (25 ml) and Sb K tartrate (0·5% soln.) (2 ml). The soln. is warmed to 70°, shaken for ½ hr. with further additions of reduced iron, cooled, filtered, and made up to 250 ml. A 100-ml portion is treated with a pinch of aluminium powder and 50 ml of conc. HCl, boiled, cooled and titrated against a standard iodine soln. C. A. SLATER

3269. **Heterometric micro-determination of lead with sodium diethyldithiocarbamate.** M. Bobtelsky and R. Rafaillof (Hebrew Univ., Jerusalem, Israel). *Anal. Chim. Acta*, 1957, **16** (4), 321–326 (in English). —From 0·02 to 0·04 mg of Pb per ml of soln. can be determined heterometrically, to within ±1%, either alone or in the presence of large excesses of Ca^{2+}, Sr^{2+}, Ba^{2+}, Mg^{2+}, Zn^{2+}, Mn^{2+}, Ni^{2+}, Al^{3+}, Cr^{3+}, Fe^{3+}, Cd^{2+}, Cu^{2+}, Sb^{3+}, Hg^{2+} and Ag^{+}. Either $Na_4P_2O_7$ or $Na_5P_3O_{10}$ must be added to complex Fe^{3+}; Bi and more than a 90% excess of Co^{2+} interfere. The sample soln. [20 ml containing *M* HNO_3 (1 to 3 ml), or *M* Na acetate (1 to 10 ml)], is titrated with 0·001 to 0·002 *M* Na diethyldithiocarbamate in the heterometer at 20° (*cf.* Bobtelsky, *Ibid.*, 1955, **13**, 172). Max. values of extinction and the sensitivity of the reaction are independent of pH; the titration curve is horizontal immediately after the first max. point. W. J. BAKER

3270. **Rapid flame-photometric determination of a micro quantity of sodium in lead metal.** Shozo Fukushima, Shiro Iwata, Sanshiro Kume and Misao Shigemoto (Chem. Dept., Fac. of Sci., Osaka Univ., Nakanoshima, Kita-ku, Osaka). *Japan Analyst*, 1956, **5** (12), 704–707.—A rapid (2 to 3 hr.) determination of Na (<0·002%) with an oxy-acetylene flame was studied. Since the intensity of the Na line is proportional to concn. for <3 p.p.m., an extrapolation method was employed. The presence of a large amount of Pb does not vitiate the estimation, provided that the same amount is added to the standard soln.; but it should be removed by pptn. as sulphate to avoid hazards that might arise in the spraying of the soln. *Procedure*—Dissolve the sample (10 g) in conc. HNO_3 (20 ml) and water (60 ml) and add dil. H_2SO_4 (1 + 3, 15·0 ml). Filter this soln. into a polyethylene bottle, dilute a 10-ml portion of the filtrate to 20 ml, and submit to flame photometry (O, 13·0 lb per sq. in., acetylene 3·5 lb per sq. in.), to give a photometer reading *R*. Mix 20 ml of Na_2SO_4 soln. (10 p.p.m.) with another 10-ml portion of the filtrate diluted to 20 ml and submit to photometry, to give a reading *R'*. The concn. (p.p.m.) of Na in the sample soln. is $2(R-R_0)/(R'-R)$, where R_0 is the intensity of the flame in the absence of Na, *i.e.*, the mean of the readings of the photometer at 580 and 600 mμ.
K. SAITO

3271. **Volumetric analysis of titanium. I.** P. Wehber, W. Johannsen and M. Heydecke (Lab. der Metallhütte Mark A.-G., Hamburg-Wilhelmsburg). *Metall*, 1956, **10** (13–14), 636–639.—A review of previous literature showed inconsistencies, particularly in respect of the reduction of Ti^{IV} soln. and the possibility of stabilising them against atmospheric or dissolved oxygen. As a preliminary to an accurate, quick and simple method for the determination of Ti, the behaviour of Ti in acid soln., particularly the redox potential, and the reduction of Ti by metals were studied. The metals studied include Pb, Cd, Zn and their amalgams, Fe and Al. These have redox potentials of < − 2 V.
G. BURGER

3272. **Determination of active titanium in titanium powder used in pyrotechnics.** M. Codell, R. P. Egan and G. Norwitz (Frankford Arsenal, Philadelphia, Pa., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (3), 228–233.—Free Ti is determined in the metal powder by dissolving a sample in a mixture of dil. HBF_4 and H_2SO_4 and measuring the volume of H evolved. The apparatus contains a magnetically operated release for dropping the sample into the acid. W. C. JOHNSON

3273. **Determination of titanium in plutonium - titanium alloys.** K. S. Bergstresser (Univ. of California, Los Alamos Lab., N.M.). *Anal. Chem.*, 1957, **29** (4, Part I), 532–534.—Addition of H_2O_2 to a $HClO_4$ soln. of the sample yields a coloured soln. with Ti and a ppt. of Pu. To a sample containing ≯ 50 mg of Pu and 50 to 500 μg of Ti, add 70% $HClO_4$ (0·25 ml). Stopper the tube with Kleenex tissue and cool; remove the stopper when the metal has dissolved. Dilute the soln. with 1·5 *N* H_2SO_4 (1 ml) and a few drops of wetting agent. Add a mixture of 1·5 *N* H_2SO_4 (1 ml) and 30% H_2O_2 (1 ml), shake and allow the mixture to stand overnight. Centrifuge, remove the supernatant liquid, and wash the ppt. with small portions of wash soln. [1·5 *N* H_2SO_4 (95 ml) plus 30% H_2O_2 (5 ml)] and dilute the combined supernatant liquid and washings to 10 ml with wash soln. Measure the extinction at 410 mμ. The standard deviation (18 results) was ± 2 μg of Ti. Interference is caused by U and Nb, but not by Th. G. BURGER

3274. **Extension of the isotopic method for the determination of oxygen in titanium.** A. D. Kirshenbaum and A. V. Grosse (Temple Univ., Philadelphia, Pa., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (3), 225–227.—The sample (1 to 2 g) of titanium metal, containing 0·04 to 0·2% of O, is mixed with ≃0·15 g of "master alloy" containing ^{18}O and is melted in a molybdenum crucible with graphite. The proportion of ^{18}O in the evolved CO is determined in the mass spectrometer, and the O content of the sample is deduced. Results on four samples are ≃10 to 13% higher than those obtained by the vacuum-fusion method. W. C. JOHNSON

3275. **Colorimetric and gravimetric determination of silicon in titanium and titanium alloys.** M. Codell and G. Norwitz (Frankford Arsenal, Philadelphia, Pa., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (4), 327–332.—In the colorimetric method, suitable for contents of Si from 0·003 to 1·5%, the sample (0·5 g) is dissolved in dil. HF, aq. 4% H_3BO_3 is added and the Ti is oxidised with 30% H_2O_2 plus 3% $KMnO_4$ (1 to 2 ml). Most of the Ti is then pptd. by heating the soln. at 100° for ≃ 90 min., and the molybdenum blue colour is then developed

in a plastics bottle at 23°. The extinction is measured on a filtered aliquot of the solution, and the Si is calculated from a calibration curve. In the gravimetric method, suitable for samples containing 0·3 to 5% of Si, the sample (1 to 2 g) is heated to fuming with H_2SO_4 and H_2O_2 (30%, 10 ml) and the soln. is cooled, diluted and filtered. The residue is ignited, and fused with Na_2CO_3; the melt is heated to fuming with $HClO_4$ (70%, 40 ml) plus H_2O (150 ml). The Ti_5Si_3 is thus completely decomposed, and the weighed ppt. of approx. pure SiO_2 can then be heated with HF and H_2SO_4, ignited at 1100° and re-weighed. The colorimetric and gravimetric results are in agreement, and the precision for each method is good. W. J. BAKER

3276. **Organic reagents for inorganic analysis. I. Phenylfluorone as sensitive reagent for colorimetric determination of zirconium.** Kenjiro Kimura and Hirotoshi Sano (Dept. of Chem., Univ. of Tokyo).. *Bull. Chem. Soc. Japan*, 1957, **30** (1), 80–83 (in English).—Phenylfluorone with a slightly acid soln. of ZrO^{2+} gives a red ppt. on standing, which, although insol. in all solvents tried, can be stabilised as a colloidal soln. by *cyclo*hexanol, and the colour measured in a spectrophotometer. To the sample soln. (< 20 ml), containing up to 50 μg of Zr, add HCl to make the final acidity 0·1 *N*, ethanol (10 ml), *cyclo*hexanol (5 ml) and ethanolic reagent soln. (0·06%) (10 ml). Make up to 50 ml with H_2O, shake, allow to stand for 2 hr. and measure the extinction at 540 mμ. The calibration curve is linear up to 0·7 p.p.m. of Zr and reproducible in the range 0·7 to 1 p.p.m. A list of interfering ions and their max. permissible amounts is given.
G. BURGER

3277. **Separation and determination of microgram quantities of zirconium.** C. E. Bricker and G. R. Waterbury (Los Alamos Scientific Lab., N.M.). *Anal. Chem.*, 1957, **29** (4, Part I), 558–562.—A method is described for the determination of Zr in plutonium alloys in the range 0·01% to 1% on samples containing 15 to 100 μg of Zr. The sample is dissolved in acid and the Zr is pptd. by addition of hydroxylamine hydrochloride and *p*-bromomandelic acid. The ppt. is separated, washed, then heated with 70% $HClO_4$ and conc. HNO_3 to destroy organic matter. The Zr is re-pptd. and dissolved in the same manner, the soln. is diluted with 2 *M* $HClO_4$, and chloranilic acid is added. The extinction at 350 mμ is measured against a blank and compared with a calibration curve. The reproducibility is within 3% when 15 μg or more of Zr is present; above 25 μg, the average recovery was 98·4%, and the standard deviation, estimated from 20 results in the presence of other ions likely to occur, is 3·4%. The double pptn. as mandelate removes most interferences. Large ratios of U give low results; Hf is assumed to interfere. A single determination takes 4 to 5 hr.; 12 can be performed simultaneously in about 6 hr.
G. BURGER

3278. **Fluorimetric determination of zirconium with morin.** R. A. Geiger and E. B. Sandell (The Univ., Minneapolis, Minn., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (4), 346–354.—Microgram amounts of zirconium can be determined fluorimetrically in 2 *M* HCl, in the presence of Al, Be, Ga, Sb, Sn, Th and U, by measuring the intensity of fluorescence (caused by an ethanolic soln. of morin) before and after the addition of EDTA, which destroys the fluorescence of Zr only. The primary filter of the photo-electric fluorimeter should transmit the 436-mμ radiation of Hg, whilst the secondary filter should have max. transmission at ≃ 525 mμ. Provided that the concn. of morin is 0·0011% when Al is present, the error in the determination of Zr is within ±5%, even when the ratio of Al to Zr is 10^4:1. Iron does not interfere if reduced to Fe^{2+} with thioglycollic acid; Ca, Mg and Ti do not interfere, and ≯0·05% of Nb or Ta should be present. The procedure is applicable to silicate rocks without any separations other than those involved in the decomposition with Na_2CO_3. An aliquot equiv. to 20 to 40 mg of the sample should be taken for the fluorescence measurements. W. J. BAKER

3279. **Spectrographic determination of lead in zircon.** N. Saillard, G. Scheibling and A. Hée. *Compt. Rend.*, 1957, **244**, 609–611.—A spectrographic technique is described in which the sample is excited in alkaline vapour; the intensity of the Pb band at 2833 A is determined and compared with a series of SiO_2 - ZrO_2 standards containing 1 to 1000 p.p.m. of Pb. S.C.I. ABSTR.

3280. **The analysis of zircon sand.** M. Akst. *Przegl. Geol.*, 1956, (4), 172–173; *Ref. Zhur., Khim.*, 1957, Abstr. No. 4820.—Mix 1 g of the ground sand with 10 g of fused NaOH, heat for 5 min. at about 600°, cool, dissolve the melt in 50 ml of hot water, add 100 ml of 20% H_2SO_4 and evaporate to dense fumes. Cool, add 20 ml of water, heat to 100°, filter off the SiO_2 and wash it with 1% H_2SO_4. After igniting and weighing the SiO_2, it is treated with HF and H_2SO_4 and the residue is dissolved in 5 ml of conc. H_2SO_4; the soln. is added to the filtrate which is made up to 500 ml. To 100 ml add 5 ml of 3% H_2O_2 and 300 ml of hot water, heat to boiling-point, precipitate Zr with a slight excess of aq. NH_3, boil for 2 min. and filter. Wash the ppt. with hot water, and transfer to a beaker with 100 ml of hot 5% HNO_3; add 70 ml of conc. HNO_3 and dilute to 300 ml. Add 5 ml of 3% H_2O_2 and 50 ml of a 1% soln. of $(NH_4)_3AsO_4$ (**I**). Heat the soln. to boiling-point, add 15 ml of a 10% soln. of **I** and boil for 20 min. Filter off the ppt. and wash it first with hot water acidified with HNO_3 and then with water; ignite to ZrO_2. Determine Fe_2O_3 and TiO_2 colorimetrically in 50-ml aliquots of the original soln. Determine Al_2O_3 on a 1-g sample of the sand; and Th and the rare earths by pptn. of 200 ml of the soln. with oxalic acid. C. D. KOPKIN

3281. **Determination of thorium and lanthanons in monazite.** J. Clinch and E. A. Simpson (Thorium Ltd., Uphall Rd., Ilford, Essex, England). *Analyst*, 1957, **82**, 258–269.—The sample (10 g) is boiled under reflux with $HClO_4$ for 3 hr. and the cooled diluted liquid is treated with HCl and hydrazine hydrochloride, the temp. being then maintained at boiling-point for 1 hr. An aliquot of the cooled filtered liquid is neutralised to the orange point of cresol red (pH 1·8) with aq. NH_3, conc. HNO_3 is added and, after further dilution, ammonium oxalate is added to the boiling soln. The paper and ppt. are treated with HNO_3 and $HClO_4$, and the pptn. and re-solution are repeated. The diluted soln. is treated with benzoic acid, and hot aq. NH_3 is added to the hot mixture to the yellow point of cresol red. The ppt., collected from the boiling liquid, is washed with boiling benzoic acid soln. and ignited and weighed as ThO_2. This filtrate is boiled and treated with aq. NH_3, and the collected ppt. is washed with ammonium benzoate soln., redissolved and re-pptd. It is then washed with very dil. aq. NH_3, ignited at 1000° and weighed as the mixed oxides. The

lanthanons occur in monazite in the tervalent state, but the ppt. contains Ce, Pr and Tb in the higher valency states. A correction is made for excess of oxygen by determining the Ce (the main oxidised component) volumetrically. A. O. JONES

3282. **Polarographic determination of ammonia and ethylenediamine.** C. J. Nyman and R. A. Johnson (State Coll. of Washington, Pullman, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 483–485.—The method depends on the anodic oxidation of mercury in the presence of ammonia (**I**) and ethylenediamine (**II**). In unbuffered 0·1 *M* KNO_3, the diffusion current (i_d) is proportional to the concn. of **II** in the range 1 to 50×10^{-4} *M*, and to the concn. of **I** in the range 1 to 15×10^{-4} *M*. In 0·1 *M* NH_4NO_3, i_d is proportional to the concn. of **I** in the range 1 to 50×10^{-4} *M*. The ratio of i_d to concn. for **I** in 0·1 *M* KNO_3 was 0·487 with a mean deviation of ±0·012 for the range 2 to 15 $\times 10^{-4}$ *M*; for **II** in 0·1 *M* KNO_3 this ratio was 0·487 ± 0·010 in the range 5 to 50×10^{-4} *M*; and for **I** in 0·1 *M* NH_4NO_3 the ratio was 0·671 ± 0·019 in the range 3 to 50×10^{-4} *M*. At lower concn., the deviation was greater. Substances that react with **I** and **II** must be excluded and also those that cause mercury to be oxidised at potentials more negative than about + 0·3 V *vs.* the S.C.E. K. A. PROCTOR

3283. **Estimation of water in fuming nitric acid.** T. B. Hoover and A. W. Hutchinson (Appl. Sci. Lab., Inc., State Coll., Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 518–522.—The sample, mixed with a soln. of H_2SO_4 in acetic acid in a conductivity cell, is titrated with a soln. of acetic anhydride in acetic acid to a break in the conductivity plot. End-points can be located with a precision corresponding to less than 0·1 mg of water. The conductivity bridge and electronic equipment are based on apparatus described by Luder (*J. Amer. Chem. Soc.*, 1940, **62**, 89). G. BURGER

3284. **Method for the determination of nitrate by titration with ferrous sulphate.** F. Krejcí and L. Kacetl. *Chem. & Ind.*, 1957, (19), 598.—A modified electrode system is described for detecting the end-point of the titration of NO_3^- by ferrous sulphate in conc. H_2SO_4. The system comprises an indicator electrode of platinum, as recommended by Strobe and Theys (*Explosifs*, 1955, **3**, 104), and a reference electrode of carbon. The applicability of this system to the titration of nitrated products as used in the explosives industry is being examined. G. S. ROBERTS

3285. **Some experiments on rate of transfer of nitrates between an aqueous and a tributyl phosphate phase.** H. A. C. McKay and D. Rees (Chem. Div., A.E.R.E., Harwell, England). A.E.R.E. C/R 1199, 1957, 8 pp.—The technique of Spence and Streeton (A.E.R.E. C/R 933, 1952) for measuring rates of transfer across an interface has been applied to transfer from tributyl phosphate (TBP) - kerosene to an aqueous phase using single rising drops of the organic phase. The apparatus and technique are described. The organic phase usually comprised HNO_3 in TBP - kerosene loaded with a small amount of the nitrate being studied. The nitrates studied were those of H, Y, Zr, Ce^{III}, Ce^{IV}, Th, U^{VI}, Np^{IV} and especially Pu^{IV}. Concn. for Th and U were 1 to 5 g per litre and for Pu about 0·1 g per litre. Counting techniques were used for most of the analyses with ^{91}Y, ^{95}Zr and ^{144}Ce as tracers. Thorium was determined by Solochrome blue (A.E.R.E. C/R 986, 1952), and U, either by the thiocyanate method (A.E.R.E. C/R 617, 1951) or by direct spectrophotometer comparison of the initial and final organic phases. Analytical errors were generally of the order of ±2%, but the authors consider the overall error in the calculation of R (rate of transfer per unit concn., *e.g.*, in g per cm² per sec. per g per litre) to be of the order of ±10%. The results for R within these limits appeared to be reproducible. For R_{Pu}, a range of $2·2$ to $3·5 \times 10^{-6}$ g per cm² per sec. per g per litre was obtained with variation of acidity and TBP concn. The general trend observed was for rates to fall with increasing atomic number. D. F. PHILLIPS

3286. **Bromimetric oxidation of red phosphorus by bromide - bromate mixtures.** M. Venugopalan and K. J. George (Hindu Univ., Benares, India). *Bull. Chem. Soc. Japan*, 1957, **30** (1), 51–53 (in English).—The use of the liberated Br from KBr + $KBrO_3$ for the oxidation of P was studied—

$$2P + 5Br_2 + 8H_2O = 12H^+ + 10Br^- + 2H_2PO_4^-,$$

excess of Br being titrated with thiosulphate. The oxidation was carried out in a stoppered flask by adding to the sample standard $KBrO_3$ soln., KBr soln. and *N* H_2SO_4 to make the total acidity 0·2 *N* H_2SO_4. The effect of temp. and concn. was studied and the energy of activation calculated. G. BURGER

3287. **The determination of phosphorus in ferrotitanium.** A. M. Dymov and R. S. Molchanova. *Primen. Radioakt. Izotop. v Metallurg., M., Metallurgizdat*, 1955, (34), 330–340; *Ref. Zhur., Khim.*, 1956, Abstr. No. 10,119.—The sample of ferrotitanium may be decomposed either by fusion with Na_2O_2, followed by leaching of the melt with hot water or NaCl soln., or by solution in a mixture of HCl and HNO_3. In the latter case, dissolve 1 g of the finely divided ferrotitanium in 15 ml of HNO_3 (1:1) and 20 ml of conc. HCl on a sand bath. After the black specks have disappeared, boil out oxides of N, cool, carefully neutralise with 25% NaOH soln. to a slight $Fe(OH)_3$ ppt. and pour into 100 ml of boiling 25% NaOH. Boil for 5 to 7 min., cool, and make up to 250 ml. Allow the ppt. to settle, and filter through a dry filter. Acidify 200 ml of the filtrate with HNO_3 (1:1) to phenolphthalein, add 5 to 8 ml of a 5% soln. of $FeCl_3.6H_2O$, and then precipitate $Fe(OH)_3$ and $FePO_4$ with 25% aq. NH_3. Filter, wash the ppt. once or twice with hot water and dissolve it in HNO_3 (1:1). Neutralise the excess of HNO_3 with aq. NH_3, add 5 ml of HNO_3 (1:1), and 8 to 10 g of NH_4NO_3, and precipitate H_3PO_4 from the hot (65° to 75°) soln. by the addition of 60 to 80 ml of $(NH_4)_2MoO_4$ soln. The determination is completed volumetrically with alkali. The presence of Si in the alloy does not cause interference. C. D. KOPKIN

3288. **Method followed in the Instituto Nacional del Carbón for the determination of phosphorus.** Anon. *Bol. Inst. Nac. Carbón*, 1956, **5** (29), 223–225.—Details are given of a standard procedure for determining P by the molybdate method. D. LEIGHTON

3289. **Coulometric titration of antimony with electrogenerated iodine.** J. J. Lingane and A. J. Bard (Harvard Univ., Cambridge, Mass., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (3), 271–273.—Quantities of Sb^{3+} from 0·06 to 10 mg are titrated in a 0·1 *M* phosphate buffer of pH 8 that is also 0·1 *M*

in KI and 0·025 *M* in K tartrate. The generator cathode is isolated from the test soln. by enclosure in a glass tube with a sintered glass bottom. Currents from 2 to 33 mA are used with titration times of 100 to 500 sec. Amperometric and potentiometric methods of end-point detection are used and errors from −0·6 to +0·8% are recorded. The visual end-point obtained with the use of starch is less precise. W. C. JOHNSON

3290. **The use of wet carbonisation in quantitative analysis. I. Determination of antimony in zinc electrolytes.** P. N. Kovalenko and N. L. Babenko. *Soobshch. Nauch. Rabot. Chlenov Vses. Khim. Obshch. im Mendeleev.*, 1955, (1), 8–11; *Ref. Zhur., Khim.*, 1956, Abstr. No. 10,096.—Antimony is determined in small quantities by the colour of a soln. of $HSbCl_6$ in benzene with methyl violet. To the soln. to be analysed add HCl to 3 or 3·5 *N*, and five drops of 2% methyl violet soln. for each 0·05 mg of Sb, extract with benzene for 3 to 5 min. and measure the extinction of the benzene layer. No interference is caused by the presence of Cd and Co (≯ 50 mg) and Fe (< 125 mg). With a concn. of Fe > 125 mg per litre, separate Sb by reduction with zinc dust or foil from 0·1 *N* H_2SO_4 (125 to 200 mg of Fe) or 0·2 *N* H_2SO_4 (200 to 1000 mg of Fe). Filter off the ppt. and excess of Zn under vacuum, treat with H_2SO_4 (1:2), add HNO_3 and tartaric acid, make up to 25 ml and determine Sb colorimetrically on an aliquot. The accuracy is within ±1·0%.
C. D. KOPKIN

3291. **Separation of bismuth from uranium using thioacetamide precipitation.** G. A. Stoner and H. L. Finston (Brookhaven National Lab., Upton, N.Y.). *Anal. Chem.*, 1957, **29** (4, Part I), 570–571.—Large amounts of Bi can be separated from microgram quantities of U by precipitating the Bi from 2 *M* HNO_3 soln. at 50° to 60° by addition of 5% thioacetamide soln. The separation was studied with the aid of Bi - U alloys enriched in ^{233}U; α counting-rate measurements showed the separation to be complete. Removal of Bi by gassing with H_2S caused loss of U. The oxychloride method failed to remove Bi completely. G. BURGER

3292. **Bismuthiol II as an analytical reagent. VIII. Volumetric determination of bismuth and palladium.** Anil Kumar Majumdar and Madan Mohan Chakrabartty (Univ. Jadavpur, Calcutta). *Z. anal. Chem.*, 1957, **156** (2), 103–105 (in English).—Bismuth is pptd. as the complex with bismuthiol II; the ppt. is dissolved in excess of EDTA (disodium salt) soln. at a pH of about 10 and the excess of EDTA is back-titrated with $MgSO_4$ soln., with Eriochrome black T as indicator. The palladium complex is pptd., dissolved in KCN soln. and the excess of KCN is back-titrated with $AgNO_3$ soln., with KI as indicator. The results for Bi are accurate within ±0·5%; those for Pd are approx. 1% low.
A. R. ROGERS

3293. **A new spot test for vanadium(V).** V. Pandu Ranga Rao and G. Gopala Rao (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1957, **156** (2), 100–102 (in English).—Mix 0·1% aq. Na salicylate (0·1 ml) with 20 *N* H_2SO_4 (2 ml) or, better, syrupy H_3PO_4 (2 ml), add the sample soln. (one drop containing ≮50 μg of V^V) and mix. A turquoise-blue colour develops at once and fades to a pale blue in 5 min. There is no interference from Fe^{III}, U^{VI}, Cr^{VI}, Mn^{VII}, Mo^{VI} or W^{VI} even in amounts of 100 μg in 2 ml; Ce^{IV} gives a black ppt. and Ti^{IV} gives a yellow colour, but these do not interfere. Ions such as Fe^{3+}, U^{4+} and Ti^{3+} interfere because they reduce the V^V. A. R. ROGERS

3294. **Hydrazinophthalazines. II. Organic reagents for the determination of vanadium.** R. Ruggieri (Military Inst. of Pharm. Chem., Florence, Italy). *Anal. Chim. Acta*, 1957, **16** (3), 246–248.—Vanadate ions yield a straw-yellow colour with hydrazinophthalazine (**I**) and dihydrazinophthalazine (**II**); **I** gives the more stable colour. The soln. for test is treated with Na acetate, adjusted to pH 5·5, **I** (0·02 g) is added and the extinction at 442 mμ is measured after 5 to 10 min. In terms of V, $E^{1\%}_{1cm}$ = 1380 to 1400. Oxidising agents, Fe, Co, Ni, Cr, Mo, W and Mn interfere.
W. C. JOHNSON

3295. **Analytical applications of complexones. VI. Spectrophotometric determination of vanadium.** F. Bermejo Martínez and A. Prieto Bouza. *Inf. Quím. Anal.*, 1957, **11** (2), 58–61.—The vanadate solution is treated with EDTA (disodium salt) (0·1 *M*) and glacial acetic acid and boiled for 10 min. On cooling, the soln. is neutralised with NaOH (2 *M*) and the absorption of the blue soln. is measured spectrophotometrically at 588 mμ (preferably within the range 0·2 to 0·7 mg of V per ml). Small amounts of W and Mo do not interfere. L. A. O'NEILL

3296. **Determination of highly dispersed carbides of vanadium, molybdenum and titanium.** N. M. Popova, A. F. Platonova, L. V. Zaslavskaya and M. F. Rŷbina. *Zavod. Lab.*, 1957, **23** (3), 269–272.—The behaviour of carbide ppt. obtained by anodic dissolution of steel is studied. The presence of surface-active substances increases the solubility of carbides in HCl soln. Molybdenum carbide, which is insol. in aq. soln. of HCl, dissolves in ethanol soln. of HCl. The use of even traces of ethanol in preliminary treatment of carbide phases is excluded if the carbides are to be separated by the acid method. G. S. SMITH

3297. **Determination of oxygen in liquids.** A. Dammers-de Klerk and B. Boot-Meurs (Univ. of Amsterdam, Netherlands). *Anal. Chim. Acta*, 1957, **16** (3), 296–297.—The method of Deinum and Dam (*Ibid.*, 1949, **3**, 353) for the determination of O in gas mixtures is adapted to the determination of O dissolved in liquids. The solubility of O in water and in 8 organic liquids is determined, each liquid being in equilibrium with air at room temp.
W. C. JOHNSON

3298. **Detection of elementary sulphur by reaction with lead thiophenoxide.** H. Krebs, H. Fassbender and F. Jörgens (Univ. Bonn, Germany). *Z. anal. Chem.*, 1957, **155** (4), 250–251.—In organic media S readily reacts with lead thiophenoxide (**I**) to form a red lead thiophenoxide polysulphide, which at room temp. is changed into black PbS. *Procedure*—Dissolve **I** (40 mg), which has been purified by pptn. from pyridine with benzene, in pyridine (5 ml). Add 5 ml of a soln. of S in benzene. With 500 μg of S a black turbidity is formed after 2 to 3 min., which rapidly settles out as a ppt. of black PbS. With 50 μg of S the turbidity appears after 1 hr. and the PbS ppt. within one day. With concn. of S of 1 to 5 μg, either a black turbidity or (dependent on the concn.) a brown to black ppt. is observed after one day. A blank test under similar conditions gives a yellow ppt. The sensitivity of the reaction may be increased at higher temp. At 70°

in the presence of 50 μg of S the turbidity appears after 5 min. and the ppt. after about 2 hr. The S must not be dissolved in CS_2 for the purpose of the test as **I** reacts with commercially pure pyridine and CS_2 to form slowly a black coloration.

D. F. PHILLIPS

3299. **Improvements in or relating to the analysis of gases containing hydrogen sulphide and sulphur dioxide.** The Gas Council (Inventors: A. B. Densham, J. D. F. Marsh and W. B. S. Newling). Brit. Pat. 769,997; date appl. 13.5.54.—The H_2S content of a gas can be determined in a continuous manner if the H_2S is converted quantitatively into SO_2, which is then measured by determining its strong and relatively specific absorption of i.r. or u.v. radiation. Oxygen (*e.g.*, as air) is added to the gas, if necessary, so that there is sufficient O present for the oxidation of the H_2S to SO_2. The gaseous mixture is passed through a first (long) cell of an analyser, the radiation absorbed in the cell being a measure of the SO_2 content of the initial gas. The gas is next passed over a hot catalyst to oxidise the H_2S to SO_2. The cooled gas is then passed into a second (shorter) cell in which the radiation absorbed is a measure of the SO_2 concn. corresponding to the concn. of SO_2 plus H_2S in the initial gas.

J. M. JACOBS

3300. **New methods of determination of binary or ternary mixtures of sulphurous and sulphuric anhydrides and of hydrogen sulphide in gaseous mixtures.** G. Pannetier, C. Meltzheim and A. Sicard (Lab. Chim. M.P.C., Fac. Sci., Dijon, France). *Bull. Soc. Chim. France*, 1956, (3), 455–459.—Methods are described for the determination of mixtures of H_2S and SO_2, SO_2 and SO_3 and of H_2S, SO_2 and SO_3, depending on titration with iodine and the determination of the acid formed, or, when all three are present, an additional oxidation with H_2O_2 in alkaline soln. and determination of the reduction in acidity, thus giving three simultaneous equations from which the constituents can be calculated.

N. E.

3301. **New methods of testing binary or ternary mixtures of sulphuric and sulphurous anhydrides and hydrogen sulphide in gaseous mixtures. Experimental procedures.** G. Pannetier and C. Meltzheim (Lab. de Chimie M.P.C., Fac. des Sciences, Dijon, France). *Chim. Anal.*, 1957, **39** (4), 142–147.—The principles underlying the methods already described (*cf. Anal. Abstr.*, 1956, **3**, 989; 1957, **4**, 3300) are briefly reviewed, emphasis being laid on experimental procedure.

W. J. WRIGHT

3302. **The analysis of sodium sulphide.** Seijiro Yoshida (Osaka Works, Sumitomo Chem. Co. Ltd., Konohana-ku, Osaka). *Japan Analyst*, 1956, **5** (12), 684–689.—Kurtenacker's method (*Analytische Chemie der Sauerstoffsäuren des Schwefels*, 1938) for the determination of oxy acids of sulphur was applied to the analysis of commercial sodium sulphide, and an industrial standard method for total S, Na_2SO_3, $Na_2S_2O_3$, NaOH, CO_3^{2-}, Na_2SiO_3 and water content is suggested.

K. SAITO

3303. **Gravimetric determination of sulphate as benzidine sulphate.** G. Gottschalk and P. Dehmel (Osram-Studiengesellschaft für elektrische Beleuchtung, Berlin). *Z. anal. Chem.*, 1957, **155** (4), 251–263.—Quantities of S in the range 0·5 to 10 mg can be determined by this method, and when it is used in combination with ion-exchange procedures interferences are eliminated and an almost universal procedure is made possible. The standard deviation based on 48 results treated in 12 groups of 4 is ±0·032 mg of S. The 99·9, 99 and 95% confidence limits are quoted as ±0·114, ±0·086 and ±0·064 mg of S, respectively. Applications of the method to specific determinations of S, such as in nickel materials, sheet iron, zirconium oxide and untreated water, are described.

D. F. PHILLIPS

3304. **Volumetric determination of sulphate by titration of excess lead nitrate with potassium chromate using siloxene indicator.** F. Kenny, R. B. Kurtz, I. Beck and I. Lukosevicius (Dept. of Chem., Hunter Coll., New York). *Anal. Chem.*, 1957, **29** (4, Part I), 543–546.—A measured vol. of standard $Pb(NO_3)_2$ soln. is added to an aq. soln. of the sample and the pH adjusted to 2·85 with HNO_3. Siloxene indicator is added and the excess of Pb is titrated with K_2CrO_4 in a dark chamber fitted to a photometer unit which measures the light emitted at the end-point by the indicator. The prep. of the indicator is described. Good results have been obtained with samples differing from standard soln. in SO_4^{2-} content by as much as 10%.

G. BURGER

3305. **Colorimetric micro-determination of sulphate by reduction to hydrogen sulphide with bivalent tin - strong phosphoric acid.** Toshiyasu Kiba and Ikuko Kishi (Dept. of Chem., Kanazawa Univ., Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (1), 44–48 (in English).—Sulphate is reduced to H_2S, absorbed in Zn acetate soln. and estimated colorimetrically by conversion into methylene blue. To the sample (containing 1 to 35 μg of S) in a quartz reaction tube add 1% $BaCl_2$ soln. (0·5 ml) and evaporate to dryness. Add 5 ml of reagent [H_3PO_4 (250 g) and $SnCl_2.2H_2O$ (50 g), boiled to expel HCl completely (*cf. Anal. Abstr.*, 1956, **3**, 2708)]. Connect an absorption vessel containing 35 ml of Zn acetate soln. (2%), pass a rapid stream of pure CO_2 through the apparatus for 5 min., then reduce the rate to one bubble per sec. and heat the reaction vessel until fumes of H_3PO_4 appear. Continue heating for 15 min. Stopper the vessel and put it in a thermostat at 24°. Add 1·5 ml of *p*-aminodimethylaniline soln. [1·4 g in 200 ml of H_2SO_4 (1 : 1)], shake, add 0·5 ml of $FeCl_3$ soln. (10%), shake and leave in thermostat for 15 min. Make up to 50 ml and measure the extinction at 655 mμ. Interferences are eliminated by a preliminary treatment of the sample with $BaCl_2$, $FeCl_3$ and aq. NH_3.

G. BURGER

3306. **Micro-determination of sodium thiosulphate by the pulse coulometric method.** M. A. V. Devanathan, Q. Fernando and P. Peries (Univ. of Ceylon, Colombo). *Anal. Chim. Acta*, 1957, **16** (3), 292–293.—The pulse coulometer (Devanathan and Fernando, *Anal. Abstr.*, 1957, **4**, 780) is used for the determination of $S_2O_3^{2-}$ or I in concn. of 50 to 100 mg per litre with an accuracy of 1%. The end-point is obtained with the use of starch as indicator.

W. C. JOHNSON

3307. **Oxidative determination of thiosulphate by alkaline ferricyanide using osmium tetroxide as a catalyst.** G. S. Deshmukh and M. G. Bapat (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1957, **156** (2), 105–108 (in English).—The catalytic action of OsO_4 has been used in the determination of thiosulphate (**I**) by titration with alkaline ferricyanide (**II**). **I** may be titrated with **II** or **II** with **I** with amperometric (dead stop) detection of end-point; the optimum KOH concn. for rapid oxidation

is 8 to 10%. If excess of **II** is added to the soln. of **I**, the ferrocyanide produced may be determined by titration with $Ce(SO_4)_2$ after acidification, or the excess of **II** may be determined by titration of the alkaline soln. with As_2O_3, or iodimetrically after acidification. A. R. ROGERS

3308. **The photometric determination of tellurium with thiourea.** W. Nielsch and L. Giefer (Anal. Lab., Joh. A. Benckiser G.m.b.H., Chemische Fabrik, Ludwigshafen/Rh.). *Z. anal. Chem.*, 1957, **155** (6), 401–406.—The colour reaction previously described (*cf. Anal. Abstr.*, 1955, **2**, 2714) has been studied with aq. H_2SO_4 or H_3PO_4 as solvent instead of aq. HNO_3. The wavelength of max. extinction is approx. 320 mμ. Reproducible results and compliance with the Beer - Lambert law are obtained with 0·2 to 8 μg of Te per ml of a soln. containing H_2SO_4 (2 to 6%) and thiourea (9 to 11%), or with 1 to 8 μg of Te per ml of a soln. containing H_3PO_4 (2 to 10%) and thiourea (10 to 12%). A. R. ROGERS

3309. **Spectrophotometric determination of tellurium.** M. W. Hanson, W. C. Bradbury and J. K. Carlton (Univ. of Arkansas, Fayetteville, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 490–491.—Soln. of Te^{IV} in conc. HCl have a yellow colour which is suitable for spectrophotometric estimation of concn. from 0·8 to 9·6 mg per 100 ml. Weigh a sample containing 1 to 120 mg of TeO_2 and make up to 100 ml with conc. HCl. Take 10 ml of this soln. and dilute to 100 ml with conc. HCl. Measure the extinction at 376 mμ against conc. HCl as blank. The calibration curve follows Beer's law up to 12 mg per 100 ml. The standard deviation calculated from 10 samples containing 5 mg of TeO_2 is ±0·01 mg. Interference is caused by Fe^{2+}, Fe^{3+}, Cu^{+}, Cu^{2+}, Ce^{4+}, VO_4^{3-}, $Cr_2O_7^{2-}$ and SeO_3^{2-} in concn. below 10 mg per 100 ml, and by Bi in concn. greater than 88 mg per 100 ml. Sexavalent Te can be reduced to Te^{IV} by boiling with HBr; tellurium metal can be dissolved in conc. HNO_3, HCl added, the soln. evaporated to dryness below 100° and the residue taken up in conc. HCl. G. BURGER

3310. **Identification of chromium.** R. Vanossi. *An. Soc. Cient. Argentina*, 1955, **160**, 37–52.—Fuse a sample containing Cr with Na_2O_2 and extract the melt with boiling water. Add Br (30 to 50 mg) and boil the soln. (for 20 to 30 sec.). Cool, and add HCl to a final concn. of 2 *N*. Cool to between 10° and 12° and extract with 2 vol. of ethyl acetate. Wash the extract with one-fifth of its vol. of cold 1 to 1·5 *N* HCl. Evaporate the extract to dryness, add a few drops of HNO_3 and again evaporate. Add more HNO_3 and $HClO_4$ and evaporate almost to dryness. Dissolve the residue in 1·5 to 2 ml of H_2O and add two drops of Br and enough NaOH to make the soln. 4 *N* in NaOH. Boil for 15 to 30 sec. and add 4 *N* $HClO_4$ until the appearance of free Br. Heat to eliminate the Br and adjust the acid concn. to 0·8 *N*. Cool to between 10° and 12° and add 0·3 to 0·4 ml of ethyl acetate, shake and add two or three drops of 3% H_2O_2 and shake again. A blue colour indicates Cr; the limits of detection are 2 to 7 μg. If no colour appears add ethyl acetate until a vol. equal to the aq. phase is present, and shake. To the ethyl acetate phase add at once 2 ml of 0·5 *N* $HClO_4$ and 0·2 ml of diphenylcarbazide (0·5% in 96% ethanol). Within a few min. a red colour will appear in the ethyl acetate layer. The limits of detection are 0·2 to 0·6 μg of Cr. In the presence of Mo, the acetate will appear pale blue or green, but will not impair the observation of a red colour due to Cr. This red colour will persist if a drop of 0·05 *M* oxalic acid is added. CHEM. ABSTR.

3311. **Photometric determination of chromium with ethylenediaminetetra-acetic acid.** W. Nielsch and G. Böltz (Laucherthal/Hohenzollern, Germany). *Metall*, 1956, **10** (19–20), 916–920.—A photometric method for the determination of Cr^{3+} depends on the formation of a violet complex with EDTA (disodium salt) soln. *Procedure*—The soln. of Cr^{3+} is boiled with a soln. of EDTA (disodium salt) (8%) for 1 min. The pH is adjusted to 3·5 and the colour developed is read at 550 mμ in a 1-cm cuvette in an Elko II spectrophotometer with an S55E filter. The method may be used for the estimation of Cr in Cr - Cu alloys. C. A. SLATER

3312. **Paper-chromatographic and paper-electrophoretic study and separation of anion-compounds of the systems molybdenum - tungsten, niobium - tantalum, and silicate - phosphate.** E. Blasius and A. Czekay (Anorg.-Chem. Inst., Techn. Univ., Berlin-Charlottenburg, Germany). *Z. anal. Chem.*, 1957, **156** (2), 81–100.—For the separation of Mo and W, paper chromatography of the oxalato complexes is recommended, with ethanol - $CHCl_3$ - 10% aq. oxalic acid (7:2:1) as mobile phase. Paper electrophoresis of the complexes in 0·1 *M* oxalic acid is also satisfactory and may be used for quant. determination. For the paper-chromatographic separation of Nb and Ta, either acetone - 2 *N* HCl - 10% oxalic acid (8:1:1) or butanone - 8 *N* HCl - 10% oxalic acid (8:1:1) is used as mobile phase; separation from Fe, V and Ti is also achieved. Satisfactory separation of sodium niobate and sodium tantalate is also obtained by paper electrophoresis in NaOH - borax buffer, but not in KOH - $KHCO_3$ buffer. Silicate and phosphate can easily be separated by paper chromatography with a soln. of borax (0·2 *M*) and HCl (0·1 *M*) as mobile phase. Electrophoresis at different pH values can be used to separate poly acids of the silicate series. A. R. ROGERS

3313. **Determination of uranium and thorium by gamma-spectrometry.** P. Keller. *Compt. Rend.*, 1957, **244**, 762–765.—The examination of the γ-ray spectrum by means of a scintillation spectrometer, which registers the low-energy γ-rays emitted, can be used to determine U and Th. Equations are developed theoretically for this purpose. S.C.I. ABSTR.

3314. **The amperometric titration of microgram and submicrogram quantities of fluoride.** J. K. Johannesson (Wellington City Council Lab., Wellington, N.Z.). *Chem. & Ind.*, 1957, (16), 480–481.—In an amperometric method for determining small quantities of F^- in materials such as food, urine and water, a standard soln. of $Th(NO_3)_4$, to which $HClO_4$ is added, is used, together with a rotating aluminium electrode and a stationary platinum anode. As little as 1·0 to 10 μg of F^- in 10 ml may be titrated, even in the presence of some $HClO_4$, which may distil over during isolation of the F^-. W. J. WRIGHT

3315. **Polarographic reduction of chlorites and bromates in anhydrous ammonia.** R. Curti and S. Locchi (Chem. Dept., Pavia Univ., Italy). *Anal. Chem.*, 1957, **29** (4, Part I), 534–537.—A quant. polarographic method is described for the determination of chlorites in the concn. range 0·004 to

0·023 *M* (sodium chlorite) and bromates in the concn. range 0·002 to 0·05 *M* (sodium bromate). A supporting electrolyte comprising 1 mole of NH_4SCN to 3 moles of liquid ammonia is used. In this medium the polarographic waves are well defined and the ratio of diffusion current to concn. is constant over the ranges quoted.

K. A. PROCTOR

3316. **Quantitative determination of water in bromine.** L. Nebbia and B. Pagani (Montecatini Res. Inst., Novara, Italy). *Chim. e Ind.*, 1957, **39** (1), 5–6.—The bromine is diluted with anhydrous CCl_4 (1:1), placed in a Drechsel bottle, dry SO_2 is passed in for 10 to 15 min. (at a rate of 0·5 to 1 litre per min.) and unchanged SO_2 is blown out with a current of argon. The CCl_4 soln. is then extracted with H_2O, the aq. extract is concentrated by evaporation in the presence of HCl, and the SO_4^{2-} are determined gravimetrically by pptn. with $BaCl_2$. A few p.p.m. of H_2O can be determined.

L. A. O'NEILL

3317. **Spectrophotometric determination of rhenium with furil α-dioxime.** V. W. Meloche, R. L. Martin and W. H. Webb (Dept. of Chem., Univ. of Wisconsin, Madison, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 527–529.—A purple-red complex of unknown composition, with an absorption max. at 532 mμ, is formed when perrhenate is reduced by $SnCl_2$ in the presence of a large excess of furil α-dioxime. To a sample containing < 300 μg of Re as perrhenate add sufficient HCl to make the final amount equal to 35 millimoles. Dilute with H_2O to about 30 ml, add 13 ml of furil α-dioxime soln. (0·70 g in 200 ml of acetone, freshly prep.) and 5 ml of $SnCl_2$ soln. (10 g dissolved in 10 ml of conc. HCl and diluted to 100 ml). Dilute to 50 ml with H_2O. After 45 min. read the extinction against a reagent blank. Molybdenum interferes and is removed by extraction of the Mo-ethylxanthate complex with $CHCl_3$.

G. BURGER

3318. **Detection of traces of iron.** F. Feigl and A. Caldas (Lab. da Produção Mineral, Min. Agric., Rio de Janeiro, Brazil). *Anal. Chem.*, 1957, **29** (4, Part I), 580–582.—When Fe^{3+} are treated with a soln. of either 2:2′-dipyridyl or 1:10-phenanthroline in thioglycollic acid, the characteristic red colour due to Fe^{2+} is produced, owing to the reduction of Fe^{3+} by the acid. In the presence of Cu, the sample soln. must be made ammoniacal before addition of the reagent. By this procedure, 0·03 μg of Fe per drop can be detected in pure iron salt soln., and 0·5 μg of Fe per drop of soln. containing 2 mg of Cu. For samples containing Cu, Ni and Co, the Fe is co-pptd. with $Al(OH)_3$ before addition of the reagent. In mineral products, including titanium white, pyrolusite and alumina, Fe is detected directly by addition of a drop of reagent to the finely ground sample on a spot plate. These methods are rapid, simple and highly sensitive.

G. BURGER

3319. **Hydrazinophthalazines. I. Organic reagents for the determination of iron.** R. Ruggieri (Military Inst. of Pharm. Chem., Florence, Italy). *Anal. Chim. Acta*, 1957, **16** (3), 242–245.—Both hydrazinophthalazine (**I**) and dihydrazinophthalazine produce red, violet or blue colours with Fe^{3+}, but **I** is preferred as a reagent for Fe^{3+} because of the greater stability of the colour it yields. The colour is developed at pH 11·2 in the presence of Na acetate and, after 15 to 30 min., the extinction is measured at 535 mμ. $E^{1\%}_{1cm} = 1233$. Oxidising agents, oxalate PO_4^{3-}, Co, Ni, Cr, Mo, W and Mn interfere.

W. C. JOHNSON

3320. **Photometric determination of iron as tributylammonium hexathiocyanatoferrate (III) and as tributylammonium ferriferronate.** M. Ziegler, O. Glemser and N. Petri (Anorg. Chem. Inst., Univ. Göttingen). *Angew. Chem.*, 1957, **69** (5), 174–177.—The advantages claimed for these methods are extreme sensitivity and freedom from interferences, *e.g.*, 2 μg of Fe can be estimated in the presence of 2 g of Al. To determine Fe as tributylammonium hexathiocyanatoferrate, mix 20 to 40 ml of sample in dil. HCl or H_2SO_4 with 2 ml of buffer soln. (pH 3·4) and 4 ml of NH_4SCN soln. (50%) in a separating funnel, add tributylammonium acetate (0·5 to 1 ml) and shake well. Extract with *iso*amyl acetate (4 ml), rinse with 1 ml and measure the absorption at 480 mμ against a blank. The colour is constant for 24 hr. The Beer-Lambert law is valid from 0 to 20 μg of Fe. To estimate Fe as tributylammonium ferriferronate, mix the sample containing up to 150 μg of Fe^{3+} in a separating funnel with 2 to 5 ml of buffer, 4 ml of ferron soln. and 1 to 2 ml of tributylammonium acetate. Extract with 5 ml of *iso*amyl alcohol and rinse with 5 ml. Measure the extinction against a blank at 610 mμ. The colour is constant for a week. The Beer-Lambert law is valid up to 150 μg of Fe. Alkali and alkaline-earth metals, Cr^{3+}, Mn^{2+} and Pb^{2+} do not interfere; other ions can be masked.

G. BURGER

3321. **Spectrochemical analysis of grey and white cast iron.** C. G. Carlsson. *Jernkontor. Ann.*, 1956, **140**, 137–147 (in English).—Direct-reading determinations of Si up to 3·5% can be made on grey or white cast iron. The same working curve can be used for both. A 220-V triggered a.c. spark was used. The fineness of the surface of the specimen must be carefully controlled, as well as its temp. A spectrograph with medium dispersion and a simple recording device can be used.

CHEM. ABSTR.

3322. **Rapid method for determining phosphorus [in iron ore].** U. Fernlund, S. Zechner and R. Olofsson. *Jernkontor. Ann.*, 1956, **140**, 386–388.—Phosphorus determinations in iron ore are made by dissolving the sample in HCl, adding $KClO_3$ and hot H_2O and pptg. as quinoline molybdophosphate. After being filtered off, the ppt. is dissolved in KOH and back-titrated with HNO_3.

CHEM. ABSTR.

3323. **Determining iron in chromium plating solutions.** H. W. Dettner. *Metal Finish.*, 1957, **55** (2), 67.—Iron present in chromium plating soln. is determined by a new complexometric procedure with EDTA (disodium salt) as chelating agent and 10% disodium catechol-3:5-disulphonate as indicator. A 5-ml aliquot of the plating soln., diluted to 150 to 200 ml, is adjusted to pH 2·5, and indicator is added to produce a blue-green colour. The soln. is titrated with 0·05 *M* EDTA (disodium salt) until the colour is just discharged.

S.C.I. ABSTR.

3324. **Use of the flame photometer for the analysis of some elements in ferrous alloys.** F. Burriel-Martí, J. Ramírez-Muñoz and M. del Carmen Asunción Omarrementería. *Inst. Hierro y Acero*, 1956, **9**, 417–424.—The response of Co, Ni, Cr, Mn and V

in the flame photometer is small and it is affected by the large amounts of Fe present. By removing the Fe, the analysis can be made, but it is not as accurate as chemical methods, although much more rapid. Errors may be caused by V, Mo and a small amount of W because they increase the flame background and have many bands in their spectra. (*Cf. Anal. Abstr.*, 1957, **4**, 124.) CHEM. ABSTR.

3325. **Fractionation method of determining hydrogen in steel.** B. A. Shmelev (Central Sci. Res. Inst. of Technol. and Engng). *Zavod. Lab.*, 1957, **23** (3), 263–269.—Vacuum extraction of H from steel can be separated into four stages corresponding to the temp. of the sample. The first fraction is liberated at room temp. or more quickly at 200°, the second at 600° to 650° with α-iron, or at 1000° to 1100° with γ-iron, the third only with molten samples at ≃1300°, and the fourth, which is rarer than the others, at 1500° to 1600°. Suitable apparatus for the fractionation method is described.

G. S. SMITH

3326. **Rapid spectrophotometric determination of copper in iron, steel and ferrous alloys.** C. U. Wetlesen (Central Inst. for Ind. Res., Oslo, Norway). *Anal. Chim. Acta*, 1957, **16** (3), 268–270.—*Procedure*—Dissolve 1 g of the iron, steel or alloy in 25 ml of H_2SO_4 (1 + 9) and 15 ml of HNO_3 (1 + 9) or, when necessary, dissolve in aqua regia and evaporate to fuming with $HClO_4$. Filter and dilute to 100 ml. To 5 ml add 10 ml of a buffer soln. containing 75 g of citric acid and 95 ml of conc. aq. NH_3 in 250 ml. Add one drop of 0·1% aq. neutral red and conc. aq. NH_3 until the indicator turns yellow; then add two to three drops of aq. NH_3 in excess. The pH is now 8·0 to 9·5. Add 5 ml of a 0·5% soln. of di-*cyclo*hexanone oxalyldihydrazone (**I**) in 50% ethanol, set aside for 2 min., dilute to 50 ml with water and measure the extinction at 595 mμ by comparison with another aliquot of the sample soln. similarly treated but with the omission of **I**. In these conditions Cu can be determined in the presence of Mn, V, Ti, Mo, W, Nb, Ta and Cr^{6+}. Large amounts of Ni and Co interfere. W. C. JOHNSON

3327. **Rapid method of determining small amounts of aluminium in steel and cast iron.** N. A. Agrinskaya (Novocherkassk Polytech. Inst.). *Zavod. Lab.*, 1957, **23** (3), 279–280.—The sample (0·05 g) of cast iron or steel is dissolved completely in 6 ml of dil. HCl (1 + 5) and the soln. is evaporated to dryness, avoiding over-heating. The residue is dissolved in water containing 0·25 ml of dil. HCl (1 + 5). The soln., filtered if necessary, is treated in a 25-ml calibrated flask with 1 ml of 10% ascorbic acid soln., 10 ml of an acetate buffer (pH 5·65), 3 ml of a 0·08% aq. soln. of stilbazo [ammonium 4 : 4′-di-(3 : 4-dihydroxyphenylazo)stilbene-2 : 2′-disulphonate] and water. The extinction is measured on a photocolorimeter with a green filter and the content of Al is read from a calibration curve. With steel containing >0·5% of Cr, calibration is carried out with soln. containing the same amount of Cr as the sample soln. The method is suitable for contents of ≃0·02%. G. S. SMITH

3328. **High-frequency combustion furnace for carbon and sulphur determinations [in iron and steel].** B. Elgh, G. Ericson and A. Wiman. *Jernkontor. Ann.*, 1956, **140**, 373–385.—The use of high-frequency heating for carbon analysis has the advantage that lumpy material can be used, saving time required for pulverising. The furnace is also instantly available and rapid determination of both C and S is possible, a total of 3 min. being required. The furnace can be used for carbon steel, alloy steel, ferro-alloys, cast iron and certain non-ferrous alloys. CHEM. ABSTR.

3329. **Determination of carbon [in iron and steel] below 0.05% by multiple factor weight combustion.** B. Bagshawe and R. H. Pinder (Brown-Firth Res. Lab., Princess St., Sheffield). Brit. Iron Steel Res. Ass. Paper MG/D/20/55, 12 pp.—To meet the increasing demand for the accurate and rapid determination of carbon in low-carbon steel and iron, the orthodox combustion procedure (B.S. 1121 : Part XI : 1948) has been modified to permit the use of larger sample weights, *e.g.*, 6 × factor weight, thereby reducing experimental error from ±0·01% to ±0·002% of C. The main requirements are: (*i*) low blank (<0·001 g of C), achieved by preheating the boats and using 7 g of prepared red lead, (*ii*), fast oxygen flow (about 2 litres per min.), (*iii*) high working temp. (about 1200°), (*iv*) a light absorption bulb (30 to 35 g). For most samples 16 g can be used without damaging the high-grade aluminous refractory tubes, but for 4% silicon-iron and for contents of C > 0·05%, charges of 11 g are advisable; for low-carbon ferrochromium, 8 g mixed with 8 g of low-carbon iron is used. Each combustion requires 10 min., so that five determinations per tube per hr. can be made when two or more tubes are in routine operation, and at least 90% of the results should lie within ±0·003% of the true figure. J. O. LAY

3330. **Spectrographic determination of lead in leaded steel.** J. E. Paterson (Graham Research Lab., Jones and Laughlin Steel Corp., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 526–527.—Direct spectrographic analysis of solid specimens of steel containing Pb in the range 0·15 to 0·35% is unsatisfactory because of lack of uniform distribution of Pb. A soln. of the sample [2 g in HNO_3 (1 : 1) (20 ml)] was therefore used, with a rotating disc electrode and excitation by a Multisource discharge. The first-order spectrum was recorded. The Pb line at 2833·07 A was used for analysis and the Fe line at 2827·9 A as internal standard. Nine determinations on NBS sample 130 (0·204% of Pb) gave a mean of 0·206% of Pb and limits of error of ±5·9% at the 95% confidence interval. G. BURGER

3331. **The spectroscopic determination of phosphorus in steel.** H. Krempl and F. Bertram (Tech. Hochschule, Munich, Germany). *Arch. Eisenhüttenw.*, 1956, **27**, 303–309.—The difficulties in determining P in steel consist mainly in the excitation of the P lines and in the disturbing lines of other elements. Several modes of excitation were tested, and the determination of P by means of the line 3175·14 A is practical. The error of this method is ±3% of the P content.

CHEM. ABSTR.

3332. **Determination of oxygen in steel by the aluminium diffusion method.** C. G. Carlsson. *Jernkontor. Ann.*, 1956, **140**, 551–574.—Several sources of error in the Gotta method (*Arch. Eisenhüttenw.*, 1943, **17**, 53), especially in killed steels and steels of high carbon content, have been eliminated by suitable changes in the method. With these changes the method is equal or superior to the vacuum-fusion method, except for speed and universality. The great sensitivity of the method

makes it possible to determine very low contents of O on small samples. CHEM. ABSTR.

3333. **Colorimetric determination of chromium in steel.** H. Carrancio de la Plaza. *Inst. Hierro y Acero*, 1956, **9**, 472–477.—The Cr in steel is oxidised to CrO_4^{2-}. The best calibration curve is obtained by adding different vol. of the dichromate soln. to pure iron samples. The presence of Fe, or time, does not influence the colour of the $Cr_2O_7^{2-}$. The best reproduction is obtained between 0·6 and 9% of Cr. When Cr is present in amounts $< 0{\cdot}25\%$ the diphenylcarbazide reaction is successful. CHEM. ABSTR.

3334. **A titration of a strong tribasic acid [hexacyanocobaltic acid] with a strong triacidic base [hexa-amminocobaltic hydroxide and tri(ethylenediamino)cobaltic hydroxide].** B. Jaselskis and H. Diehl (Iowa State College, U.S.A.). *Anal. Chim. Acta*, 1957, **16** (3), 274–275.—A soln. ($\simeq 0{\cdot}18\ N$) of hexa-amminocobaltic hydroxide is prepared by passing a soln. of the chloride through a column of Amberlite IR-410 anion-exchange resin. A soln. ($\simeq 0{\cdot}09N$) of tri(ethylenediamino)cobaltic hydroxide is prepared similarly from the bromide of this base. Both these soln. are used to titrate a soln. ($\simeq 0{\cdot}17N$) of hexacyanocobaltic acid prepared by passing a soln. of its potassium salt through a column of Amberlite IR-120 cation-exchange resin. The potentiometric titration curves are typical of those obtained when a strong acid is titrated with a strong base. W. C. JOHNSON

3335. **Micro-analysis with the aid of ion-exchange resins. XI. Detection of small amounts of nickel with dimethylglyoxime and bromine water.** Masatoshi Fujimoto (Chem. Inst., Fac. of Science, Tokyo Univ., Japan). *Bull. Chem. Soc. Japan*, 1957, **30** (1), 93–96 (in German).—The pink or brown colour given by ammoniacal aq. soln. of nickel salts in the presence of oxidising agents with dimethylglyoxime is greatly intensified by adsorption on an anion-exchange resin. Place one drop each of sample soln., 1 *N* aq. NH_3 and 0·3% bromine water together on a white spot-plate. If the concn. of salts in the sample exceeds 0·3% add 1 ml of H_2O. After 5 min. add one drop of 1% ethanolic dimethylglyoxime soln. and a few grains of resin (Dowex 1-X4, RCl form), stir and after 20 min. examine the resin with a lens for an orange - pink colour. Only Fe^{3+} and Fe^{2+} interfere significantly. The limit of detection is $0{\cdot}011\ \mu g$ in a limiting concn. of 1 in $3{\cdot}8 \times 10^6$. G. BURGER

3336. **Colorimetric determination of ruthenium.** S. B. Knight, R. L. Parks, S. C. Leidt and K. L. Parks (Univ. of North Carolina, Chapel Hill, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 571–574.—Of the six sulphur-containing compounds tested, *sym.*-diphenylthiourea was found to be the most suitable reagent owing to the stability of the colour of the complex it forms with Ru and its specificity for Ru. *Procedure*—The sample soln. (10 ml) in 6 *N* HCl is mixed with a soln. (1:1) of conc. HCl in ethanol (30 ml), and a 2% soln. of the reagent in glacial acetic acid (5 ml) is added. The soln. is diluted to 45 ml with the HCl - ethanol soln., heated at 85° for 5 min., cooled, and diluted to 50 ml, and the extinction at 630 mμ is measured. Blanks are prepared in the same manner. Reproducibility of results was good and the optimum concn. range was 6 to 18 p.p.m. of Ru. If foreign ions are present in excess, the Ru should first be separated as the tetroxide. Conditions and results for the other reagents are also reported. G. BURGER

3337. **Use of organic reagents in inorganic analysis. IV. Determination of palladium and uranium with 2-hydroxy-1-nitroso-3-naphthoic acid.** Sachindra Kumar Datta (Gov. Coll., Darjeeling, India). *Z. anal. Chem.*, 1957, **155** (4), 241–250 (in English).—The pH ranges for complete pptn. of Pd and U with this reagent are from 2·6 to 4·8 and from 3·4 to 4·5, respectively. Separation of Pd and U from Th and Zr depends on the fact that both the palladium and uranium complexes are sol. in dilute aq. NH_3 without decomposition. Separation from Co is achieved by extraction of the cobalt complex with *iso*butyl methyl carbinol. Results indicate that up to 4 mg of Pd or U may be determined and that the method is stoicheiometric within this limit. For gravimetric determination the palladium complex was dried to constant weight at 130° and then ignited to metal; the uranium complex was dried to constant weight at 120° and then ignited to U_3O_8. It has not been possible to separate Pd from U by means of this reagent. D. F. PHILLIPS

3338. **Quantitative differential thermal analyses of clay and other minerals.** H. W. van der Marel. *Amer. Mineral.*, 1956, **41**, 222–244.—The shape and intensity of the thermal curve of minerals, when analysed by the differential thermal analysis (D.T.A.) method, are strongly influenced by amorphous coatings and disordered structures on the surface of the particles (Beilby layer) and also by differences in particle and crystallite size, degree of crystallinity of the crystallites and ion substitution in the crystal structure. Consequently, accurate quant. determination of the amount of a clay mineral by D.T.A. is impossible. This conclusion is based on experiments where errors due to the D.T.A. apparatus are eliminated by calibration with a standard chemical, use of the same sample holder and thermocouple, dilution of the mineral with the inert reference material and packing the mixtures always in the same way in the sample holder. BRIT. CERAM. ABSTR.

3339. **Conductimetric determination of alkalinity in red drilling muds.** M. L. Bordera and A. Molinari (Ist. Chim. Gen. Appl. Metallurg., Politecn., Turin). *Ric. Sci.*, 1957, **27** (1), 92–99.—The rapid method described is based on the conductimetric titration of the fluid with oxalic acid. In this way, separate determination of $Ca(OH)_2$ and NaOH is possible, without filtration of the mud. The results are compared with those of other techniques, and the effect of foreign ions on the conductimetric curve is discussed. C. A. FINCH

See also Abstracts 3210, 3211, 3212, 3213, 3215, 3219, 3226, 3521, 3522.

3.—ORGANIC ANALYSIS

3340. **Use of mass spectrometry in organic analysis.** E. L. Eliel, T. J. Prosser and G. W. Young (Univ. of Notre Dame, Ind., U.S.A.). *J. Chem. Educ.*, 1957, **34** (2), 72–77.—A review is presented of the literature dealing with mass-spectrometric techniques for analysis of org. compounds of mol. wt. < 600 and of appreciable volatility. (36 references.) S.C.I. ABSTR.

3341. **Direct titration of organic compounds with chromous sulphate.** H. Jucker (Ciba A.-G., Basle, Switzerland). *Anal. Chim. Acta*, 1957, **16** (3), 210–213.—Various nitroso, nitro and azo compounds and some quinones are titrated with 0·1 *N* $CrSO_4$. The samples (50 to 500 mg) are dissolved in water, dimethylformamide, pyridine or dil. H_2SO_4, the soln. is diluted to 50 ml with *N* H_2SO_4 and the course of the titration is followed potentiometrically by means of a platinum electrode and a calomel reference electrode. Certain *o*- and *p*-isomers yield separate inflections and are titrated individually in the same soln. W. C. JOHNSON

3342. **The volumetric micro-determination of chlorine, especially in organic substances.** W. Pilz (Org. anal. Lab., Farbenfabriken Bayer A.-G., Leverkusen). *Z. anal. Chem.*, 1957, **155** (6), 423–428.—In this determination of small quantities ($\simeq$ 0·5 mg) of Cl in organic substances or in large volumes ($\simeq$ 1000 ml) of soln., the Cl is first brought into the ionic state and pptd. completely with excess of $AgNO_3$. The ppt. is washed, dissolved in aq. NH_3 and re-pptd. by aq. H_2SO_4, and the colloidal suspension of AgCl is titrated with 0·01 *N* KI. The end-point is detected by a modification of the starch - iodine reaction of Kainz and Resch (*Mikrochim. Acta*, 1952, **39**, 292). The results of 21 recovery experiments indicated that the error is usually $< \pm 0{\cdot}5\%$. No interference was shown by 15 substances in the concn. likely to be present in samples. A. R. ROGERS

3343. **Rapid method to determine sulphur.** R. Ottosson and O. Snellman (Inst. of Biochemistry, Univ. of Uppsala, Sweden). *Acta Chem. Scand.*, 1957, **11** (1), 185–187.—Some modifications have been introduced into Paulson's method for determining S in organic compounds (*Brit. Abstr. C*, 1953, 360). A 500-ml bottle with ground stopper is used for the combustion, and platinum wire, passing through the stopper, terminates in a loop into which the sample in a filter-paper strip is inserted. The sample is burned by Schöniger's method (*cf. Anal. Abstr.*, 1955, **2**, 1816) but without using NaOH. Water (10 ml) and 30% H_2O_2 (2 ml) are added to the bottle, and O is passed in for $\simeq$20 sec. The paper strip is ignited, and the bottle left for 30 min. After rinsing the ground surfaces, 10 ml of the reaction mixture is put in the conductivity cell with one drop of 20% acetic acid and 1 ml of an aq. $BaSO_4$ suspension. After 10 min., the apparatus is adjusted to zero deflection and the resistance lowered about 40 ohms. The titrating soln. (0·5 *M* $BaCl_2$) is used in 0·002-ml portions. Readings are plotted against total displacement of the micrometer screw. Sulphanilamide was used for calibration, and calibration curves are shown for the regions 100 to 250 μg and 20 to 80 μg. W. J. WRIGHT

3344. **High results in methoxyl determinations on mixtures containing polyhydric alcohols.** E. von Rudloff (Nat. Res. Council of Canada, Prairie Regional Lab., Saskatoon, Canada). *Anal. Chim. Acta*, 1957, **16** (3), 294–295.—Ethanediol and propane-1:2-diol, when treated by the Zeisel method for methoxyl determination, yield ethyl iodide and a smaller amount of vinyl iodide, producing apparent methoxyl values between 26 and 35%. Sorbitol, mannitol, butane-2:3-diol and glycerol also give methoxyl values ranging from 5 to 26%. W. C. JOHNSON

3345. **Nitrometry of methoxyl group.** Kiyoshi Takiura, Yoshio Takino and Shihoko Harada (Pharm. Fac., Osaka Univ., Hotarugaike, Toyonaka, Osaka). *J. Pharm. Soc. Japan*, 1956, **76** (11), 1328–1330.—The sensitivity of the determination of methoxyl groups by Elek's method is increased by the use of nitrometry in place of iodimetry at the final stage. Methyl iodide is absorbed in a mixture of Br and acetic acid in a stoppered glass tube, and made up to 20 ml with 50% acetic acid (5 ml) and 20% Na acetate soln. An aliquot is treated with formic acid (a few drops) to reduce Br and submitted to nitrometry with KI in saturated NaCl soln. (6 ml) and hydrazine. The presence of an excess of formic acid results in low values, owing to the reduction of HIO_3. K. SAITO

3346. **Detection of the epoxide group by infra-red spectroscopy.** H. B. Henbest, G. D. Meakins, B. Nicholls and K. J. Taylor (Univ., Manchester, England). *J. Chem. Soc.*, 1957, 1459–1462.—The i.r. spectra of some epoxides was studied in the 3200 to 2800-cm^{-1} region. Compounds having a 1:2-disubstituted epoxide group attached to an acyclic nucleus or to a *cyclo*hexane ring showed a separate peak at approx. 3000 cm^{-1}. The main C–H stretching bands occur at 2958 to 2580 cm^{-1}. When the epoxide group is fused to a 5-membered ring the bands appear at approx. 3035 cm^{-1}. When the main C–H bands are very intense, as in steroids, the epoxide bands cannot be detected. The frequencies and intensities of the epoxide bands and main C–H bands of the compounds studied are presented. G. BURGER

3347. **New approach to the determination of microgram quantities of phosphorylating or acylating agents.** B. Saville (Chem. Defence Exp. Estab., Porton, Wilts., England). *Analyst*, 1957, **82**, 269–274.—In the interaction of *iso*nitrosoacetone (**I**) with phosphorylating and acylating agents, *viz*, phosphofluoridates, tetra-alkyl pyrophosphates, acetic anhydride and benzoyl chloride, the cyanide ion is formed in amount equiv. to the original agent present. The agent in a suitable water-miscible solvent is mixed with **I** in a borate buffer soln. and the reaction is allowed to proceed long enough to ensure formation of the total equiv. of CN^-. Chloramine T is added and then a pyridine - pyrazolone reagent (prep. described) to determine the CN^-, the intensity of the blue colour being measured in a Spekker absorptiometer and referred to a calibration graph. Details are given of the application of the method to the determination of Sarin (*iso*propyl methylphosphonofluoridate). The only variation for application to different samples is the time necessary for complete formation of the equiv. of CN^-. These times are quoted for some samples and may be found for others by a preliminary expt. Free halogens interfere and mercuric salts and large amounts of peroxides oxidise CN^- to CNO^-. Common inorganic ions do not interfere. A. O. JONES

3348. **Determination of hydrogen in mine airs using a gas-chromatographic technique.** P. Whatmough (Central Lab., N.W. Div., Nat. Coal Bd., Manchester, England). *Nature*, 1957, **179**, 911–912.—Hydrogen and methane are determined by gas chromatography, with a 7-ft. activated carbon column and a platinum-wire katharometer. G. SKIRROW

3349. **Spectrophotometric titration of olefins with electrically generated bromine.** J. W. Miller and D. D. DeFord (Northwestern Univ., Evanston, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 475–479.—The titrations are carried out in a mixture of glacial acetic acid and methanol (3:1), in the presence of a small quantity of KBr and HCl; $HgCl_2$ is used as a catalyst for direct titrations. The use of the spectrophotometric end-point has the advantages—(*i*) improved selectivity and variable sensitivity, and (*ii*) the exact end-point in titrations that are slow in the vicinity of the end-point can be determined by extrapolation of the linear portion of the titration curve before and after the end-point. The bromine numbers of 13 olefins of various structures were determined with a precision better than ±2% on samples ranging from 3·4 to 11 mg. G. P. Cook

3350. **Hexachloroethane.** British Standards Institution (2 Park St., London). B.S. 577 : 1957, 10 pp.—This is a revision of the 1951 standard. The limits for three impurities have been reduced, *viz*, alcohol-insol. matter to 0·05%, water-soluble chloride to 0·01% as NaCl, and alkalinity to 0·01% as Na_2CO_3. This has enabled the ash limit to be reduced to 0·03%. G. S. Roberts

3351. ***iso*Propyl alcohol.** British Standards Institution (2 Park St., London). B.S. 1595 : 1957, 9 pp.—This is a revision of the 1950 standard. Specific gravity limits at 25°/25° have been set at 0·782 to 0·786, and a limit has been introduced for water, determined by the Fischer method, at ⩾0·5% by wt. A procedure for testing the miscibility with water is now included. G. S. Roberts

3352. **Simultaneous detection, identification and determination of secondary and tertiary alcohols on a micro scale.** M. Jureček, O. Chládek, R. Chládková, M. Souček and B. Srpová (Inst. Anal. Chem., High School Chem. Technol., Pardubice, Czechoslovakia). *Chem. Listy*, 1957, **51** (3), 448–451.—Alcohols can be converted by means of Lucas's reagent [(*a*) conc. HCl, (*b*) 136 g of anhyd. $ZnCl_2$ in 105 g of conc. HCl] into the corresponding alkyl chlorides, which can be isolated and converted by reaction with thiourea into soluble alkylthiuronium chlorides which yield with Na 3:5-dinitrobenzoate an insol. addition product. This derivative can be identified by its m.p. and the nitro group can be determined by titration with $TiCl_3$. J. Zýka

3353. **Diacetone alcohol.** British Standards Institution (2 Park St., London). B.S. 549 : 1957, 7 pp.—This is a revision of the 1950 standard. The chief modifications are that the distillation-range test is now regarded as being unreliable and will no longer be used; the minimum flash-point has been raised to 125° F; specific gravity limits at 25°/25° have been set at 0·932 to 0·938; a limit for water has been set at ⩾0·50% by wt.; and a determination of n_D^{20} (1·4225 to 1·4245) has been introduced. A procedure is described for testing the miscibility of the material with water. G. S. Roberts

3354. **A polarographic method for the study of glycol fission by periodic acid.** P. Zuman and J. Krupička (Polarogr. Inst., Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1957, **51** (3), 424–432.—The polarographic determination of periodic acid by means of its reduction wave is suitable for the study of the reaction of periodate with glycols. The advantage of this method is the velocity (which allows the kinetic analysis of the reaction), the specificity, and the possibility of the analysis on a micro scale. This method was used for the analysis of the mixture of threo- and erythro-epimers of open-chain 1:2-diols and is suitable also for the determination of total amount of periodic acid consumed in structural studies. J. Zýka

3355. **Derivative polarography of carbohydrates. The aldopentose hydrazones.** J. W. Haas, jun., and C. C. Lynch (Univ. of Delaware, Newark, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 479–481.—Aldopentoses in the concn. range 1×10^{-3} to 2×10^{-2} *M* can be qual. and quant. determined polarographically by introducing the sugar into a buffered hydrazine sulphate soln. of pH 2·3. A single well-formed hydrazone wave is obtained, and reduction currents are directly proportional to concn. Sugars can be determined singly and in pairs with an error of <2% for the individual sugars, and from 2 to 10% for the pairs under certain limiting conditions. K. A. Proctor

3356. **Application of anthrone test to determination of cellulose derivatives in non-aqueous media.** E. P. Samsel and J. C. Aldrich (Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 574–576.—Ethylcellulose is extracted from light mineral oil with a methanol - water solvent, and quant. determined by a modified anthrone method. Cellulose acetate butyrate is determined similarly, but a methanol - acetone solvent mixture is used for the extraction. Essentially all of the cellulose derivative content of mineral oil was removed by both extraction procedures at various concn. levels. The accuracy of the anthrone method was within 1 p.p.m. G. P. Cook

3357. **Acetone.** British Standards Institution (2 Park St., London). B.S. 509 : 1957, 9 pp.—This is a revision of the 1950 standard, the main modification being that the length of time allowed for decolorisation in the permanganate test has been increased to 120 min. Specific gravity limits at 25°/25° of 0·786 to 0·788 have been introduced; a method is now described for testing miscibility with water; and a limit for water of ⩾0·7% by wt. has been set. G. S. Roberts

3358. **Quantitative determination of maleic anhydride and maleic acid.** L. Nebbia and F. Guerrieri (Montecatini Res. Inst., Novara, Italy). *Chim. e Ind.*, 1957, **39** (1), 17–18.—Maleic anhydride or maleic acid (1 to 1·5 g) in the presence of other dibasic acids and anhydrides is treated with anthracene (2 to 2·5 g) and chlorobenzene (10 ml) and heated under reflux for 0·75 hr. Boiling H_2O (100 ml) is added with shaking for 1 to 2 min. and, on cooling, the mixture is titrated with 0·5 *N* NaOH. The adduct of anthracene with maleic anhydride or maleic acid is insol. in H_2O and cannot be titrated under these conditions, whereas other acids and anhydrides can be titrated completely. From the reduction of acidity after reaction with anthracene the amounts of maleic anhydride or maleic acid may therefore be calculated. The method may be applied in the presence of phthalic anhydride, tartaric acid or succinic acid. If tetrahydrophthalic anhydride or fumaric acid is present the chlorobenzene must be steam-distilled off before titration. Ketones and aldehydes do not interfere. The accuracy is ±0·3%. L. A. O'Neill

3359. **Critical studies of micro-determination of citric acid by the pentabromoacetone method.** K. Täufel and H. Ruttloff (Inst. f. Ernährungsforsch. u. Anstalt f. Vitaminforsch., Potsdam-Rehbrücke, Germany). *Ernährungsforschung*, 1956, **1** (4), 693–703.—Modifications of the colorimetric method of Pucher *et al.* are described. Results obtained are affected by $>1\%$ by colour-fading, provided that determinations are carried out in diffuse daylight at $>20°$ and (especially) provided that spectrophotometric measurements are made after an interval of >15 min.; the keeping of the coloured solutions in darkness before spectrophotometric measurement is recommended. As a colour stabiliser, 50% glycerol (freshly distilled) is more effective than pyridine or ethanediol, but under suitable conditions the use of a stabiliser is not strictly necessary. P. S. ARUP

3360. **Method for the determination of acetic anhydride in mixtures with acetic acid.** T. Ellerington and J. J. Nicholls (British Industrial Solvents, Hedon, Hull, England). *Analyst*, 1957, **82**, 233–237.—In the method described, acetic anhydride is allowed to react with excess of aniline and the uncombined aniline is determined by titration with $HClO_4$ in glacial acetic acid soln. The end-point is determined either potentiometrically with use of a glass indicator electrode and a saturated calomel reference electrode with a high-impedance millivoltmeter, or visually by use of crystal violet as indicator. The colour indicating the correct visual end-point varies with the sample being titrated, and a method for ascertaining the behaviour of the indicator at the end-point with a particular sample is described. A suitable cell for the potentiometric titration is described and illustrated, and directions are given for the preparation and standardisation of the reagents. A. O. JONES

3361. **Colorimetric determination of acetic anhydride and related compounds.** H. F. Liddell and B. Saville (Chemical Defence Exp. Estab., Porton, Wilts.). *Chem. & Ind.*, 1957, (16), 493–494.—Certain primary aromatic amines react with mixtures of acetic anhydride and nitrite to form diazotates, which may be coupled with alkaline aminonaphtholsulphonic acids to yield intense water-soluble dyes. In a method for determining microgram quantities of acetic anhydride, the reagents are—(*a*) a soln. of 80 ml of *N* Na acetate and 100 ml of acetone, in which is dissolved 0·2 g of Brentamine Fast Violet B base (3-amino-4-methoxy-6-benzoylaminotoluene); 20 ml of *N* aq. $NaNO_2$ is added; (*b*) 0·05% "2S acid" (8-amino-1-naphthol-2:4-disulphonic acid) in 1% aq. Na_2CO_3 soln. To reagent (*a*) (5 ml) add 0·5 to 2·0 ml of an acetone soln. containing 5 to 100 μg of acetic anhydride. Set aside for 5 min., make up to 25 ml with reagent (*b*), and measure the extinction. For 0·2 to 2 mg of acetic anhydride, the acetone soln. of the anhydride is mixed with a mixture of aq. $NaNO_2$, aq. Na acetate, and diphenylamine in acetone. The accuracy is within $\pm 2\%$. W. J. WRIGHT

3362. **Ethyl lactate.** British Standards Institution (2 Park St., London). B.S. 663 : 1957, 9 pp.—This is a revision of the 1950 standard. Specific gravity limits at 25°/25° have been set at 1·028 to 1·034, and a limit for water, determined by the Fischer method, has been set at $>0·70\%$ by wt. The minimum ester content has been raised from 97% to 98%. G. S. ROBERTS

3363. **Amyl acetate.** British Standards Institution (2 Park St., London). B.S. 522 : 1957, 9 pp.—This is a revision of the 1950 standard. Specific gravity limits at 25°/25° of 0·864 to 0·872 have been introduced, and a limit for water, determined by the Fischer method, of $>0·3\%$ by wt. has been set. G. S. ROBERTS

3364. **A new reaction for amines and its use in qualitative and quantitative analysis.** N. V. Reis. *Sb. Nauch. Trudy Samarkandsk. Med. Inst.*, 1956, **11**, 117–124; *Ref. Zhur., Khim.*, 1957, Abstr. No. 4848.—The reaction of NaBrO with primary, secondary and tertiary amines results in the formation of bromamines, which react with KI with liberation of iodine in an amount equiv. to that of the amine. This is the basis of a method for the qual. and quant. determination of amines. The error of the determination is $>6\%$. The high sensitivity of the reaction enables it to be used as a micro-analytical method. C. D. KOPKIN

3365. **Determination of water in several hydrazines.** H. F. Cordes and C. W. Tait (U.S. Naval Ordnance Test Station, China Lake, Calif., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 485–487.—The 1·9-μ absorption band of water is used for its determination in hydrazine, methylhydrazine and 1:1-dimethylhydrazine. Beer's law is obeyed over the range 0·1 to 15% by wt. and a temp. variation of 2° in the soln. has little effect on the result. Dimethylamine and dimethylnitrosamine do not interfere seriously, but methanol and ethanol do so if present in concn. $>1\%$ by wt. G. P. COOK

3366. **Estimation of melamine in presence of guanidine.** R. M. Engelbrecht, H. E. Moseley, W. P. Donahoo and W. R. Rolingson (Monsanto Chemical Co., El Dorado, Ark., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 579–580.—Three gravimetric methods are described. (*i*) Melamine is pptd. under acid conditions, as the picrate in the presence of guanidine; the temp. of the pptn. is controlled. (*ii*) The guanidine is eliminated by pptn. as the picrate under alkaline conditions and the melamine is then determined as the picrate in acid media. (*iii*) The melamine is determined by extraction with conc. NaOH soln. Reasonable agreement was attained between the methods. G. P. COOK

3367. **Determination of melamine by precipitation with cyanuric acid.** L. Nebbia, F. Guerrieri and B. Pagani (Montecatini Res. Inst., Novara, Italy). *Chim. e Ind.*, 1957, **39** (2), 81–83.—A neutral soln. of melamine ($\simeq 0·2\%$) in the presence of K acetate is treated with a soln. of cyanuric acid ($\simeq 0·1\%$). The ppt. of melamine cyanurate is filtered off, dried at 105° to 110° and weighed. Alternatively, the soln. of melamine may be titrated with the cyanuric acid soln. and the end-point determined conductimetrically. A melamine content of 5 to 50 mg may be determined within $\pm 0·1\%$ gravimetrically or $\pm 0·3\%$ conductimetrically. Ammeline, ammelide or urea do not interfere. Conversely, cyanuric acid may be determined. L. A. O'NEILL

3368. **Quantitative spectrophotometric determination of 6-hydroxypurine in the presence of 2:6-dihydroxypurine.** S. Tricerri and A. Braschi. *Il Farmaco*, 1957, **12** (1), 28–33.—A rapid method of determining 6-hydroxypurine (hypoxanthine) in the

presence of 2:6-dihydroxypurine (xanthine) is suggested by plotting a straight line $R = f(x)$ in which R represents the ratio of optical densities and x the relative concn. of hypoxanthine. Provided the line is plotted on a sufficiently large scale to permit an accurate reading of the spectrophotometric data, the resulting errors are $< \pm 0{\cdot}4\%$. The method is based on a suggestion of Bonnier and Gaudemaris (*Anal. Abstr.*, 1955, **2**, 2322) for the determination of a mixture of ethanol and cinnamaldehyde, which is discussed. P. Haas

3369. **Colorimetric micro-method for quantitative determination of pentachlorophenol.** J. Erkama and A. Laamanen (Biochem. Lab., Univ. of Helsinki, Finland). *Suomen Kem.*, 1956, **13**, 37–39 (in English).—Acidify the soln. containing < 2 mg of pentachlorophenol (**I**) with 10% HCl and extract twice with ether. Evaporate the ether, add conc. HNO_3 (0·5 ml) to the residue and heat in a boiling-water bath for 1 min. Cool and add saturated urea soln. (0·04 ml), mix, add 0·5% ethanolic tetramethyl-*p*-diaminodiphenylmethane soln. (0·2 ml), shake thoroughly and add saturated Na acetate soln. (1 ml). Dissolve the blue-green ppt. by adding 5% acetic acid (4 ml) and measure the extinction at 600 mμ. The method is reliable for 0·1 to 1·5 mg of **I**. H. F. W. Kirkpatrick

3370. **Diethyl phthalate.** British Standards Institution (2 Park St., London). B.S. 574 : 1957, 8 pp.—This is a revision of the 1950 standard. Specific gravity limits at 25°/25° have been set at 1·115 to 1·119, and a limit for water, determined by the Fischer method, has been set at $\geqslant 0{\cdot}20\%$ by wt. For some of its uses the odour of the material is important and it is recommended that "agreed samples" should be adopted by interested parties when necessary. G. S. Roberts

3371. **Oxidimetric estimation of 4:4′-dihydroxydiphenyl.** M. Matrka and F. Navrátil (Research Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1957, **7** (3), 136–137.—A rapid method for the determination of 4:4′-dihydroxydiphenyl with tervalent copper soln. is described. *Procedure*—Dissolve the sample (1 g) in hot water (100 to 150 ml) and *N* NaOH (2·5 ml) and dilute to 250 ml. To a 20-ml aliquot add water (80 ml) and an excess of Na_2HPO_4 and titrate potentiometrically with 0·1 *M* potassium diperiodatocuprate^{III}. J. Böswart

3372. **Analysis of the terphenyls by a compressed pellet infra-red method involving micro-distillation.** E. L. Colichman, R. F. Fish and G. O. Bjarke (N. American Aviation, Inc., Canoga Park, Calif., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (3), 250–258.—Radiation-damaged samples of the terphenyls are first separated from higher-boiling materials by vacuum distillation on a micro scale; the terphenyl is then determined by i.r. absorption by the compressed pellet (KBr) method (*cf.* Stimson and O'Donnell, *Brit. Abstr. AI*, 1952, 765). W. C. Johnson

3373. **The use of polarographic analysis in the determination of 2:3:5:6-tetrachloronitrobenzene in a technical product.** J. Kováč (Res. Inst. Agrochem. Technol., Bratislava, Czechoslovakia). *Chem. Zvesti*, 1956, **10** (7), 430–435.—A polarographic determination of 2:3:5:6-tetrachloronitrobenzene (**I**) in a technical product was carried out in a supporting electrolyte containing 0·2 *M* Na acetate and 0·2 *M* acetic acid mixed with *iso*propyl alcohol (1:1). The sample must be dissolved in acetone. The $E_{\frac{1}{2}}$ of **I**, 2:3:4:5-tetrachloronitrobenzene, 2:3:4:5:6-pentachloronitrobenzene and 2:4:5-trichloronitrobenzene were measured and the influence of these components on the determination of **I** in a technical product was studied. J. Pelikán

3374. **Paper chromatography of 2:4-dinitrophenylhydrazones of alka-2:4-dienals.** D. A. Forss, E. A. Dunstone and W. Stark (C.S.I.R.O., Melbourne, Australia). *Chem. & Ind.*, 1957, (17), 521.—The R_F values of the 2:4-dinitrophenylhydrazones of some saturated aldehydes and ketones, alk-2-enals and alka-2:4-dienals are tabulated. The presence of double bonds in the 2- and 4-positions in a saturated aldehyde lowers the R_F value to that of the saturated aldehyde with three fewer C atoms. G. P. Cook

3375. **Infra-red analysis of toluene-2:4-di*iso*cyanate and toluene-2:6-di*iso*cyanate mixtures.** S. S. Lord, jun. (E. I. du Pont de Nemours & Co., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 497–499.—Two procedures are described: one for analysing mixtures in which the 2:4-isomer content is between 5 and 95%, and the other when the 2:4-isomer content is >95%. Both methods are based on the quant. measurement of bands at 12·35 and 12·8 mμ arising from C–H deformation vibrations of the aromatic ring. The 2:5-isomer interferes slightly with the 2:4-isomer determination, but it is not normally present in these mixtures in sufficient quantity to give serious errors. The precision of the 2:4-isomer determination was ±0·8%, absolute, for the mean of duplicate determinations, and the precision for the 2:6-isomer determination was ±0·08%, absolute, at the 1·5% level. G. P. Cook

3376. **Potentiometric determination of tetraphenylboron with silver nitrate.** F. E. Crane, jun. (Rutgers Univ., New Brunswick, N.J., U.S.A.). *Anal. Chim. Acta*, 1957, **16** (4), 370–377.—The content of $(C_6H_5)_4B^-$ in organic amine tetraphenylboron derivatives can be determined rapidly and accurately by potentiometric titration with $AgNO_3$. The dry sample (60 to 150 mg) is dissolved in aq. acetone (1 + 1) and the soln. is buffered at pH 5 with 3 *M* acetic acid (5 ml) and 3 *M* Na acetate (5 ml). The resulting soln. (≃ 80 ml) is then titrated with 0·06 *N* $AgNO_3$, the Sargent-Malmstadt automatic titrator being used, with a platinum indicator-electrode and a shielded platinum reference-electrode. End-points are sharp except for the primary aromatic amine compounds. The error is within ±2% (analyses of 22 different compounds). The $AgNO_3$ soln. must be standardised against Na or K $B(C_6H_5)_4$. W. J. Baker

3377. **Colour reactions of iminazoline compounds.** Shintaro Kikuchi (Pharm. Dept., Sapporo Medical College, Minami 1-jo, Sapporo). *J. Pharm. Soc. Japan*, 1956, **76** (11), 1290–1292.—Iminazoline compounds (>50 μg per ml) react with Na pentacyanoamminoferroate (**I**) (0·5% aq. soln.), in the absence of free acid or base, to produce a blue-violet coloration. Iminazole or purine derivatives are inactive towards **I**, whilst thiocarbazole compounds, hydrazine, sodium azide and some nitroso compounds undergo a similar reaction. A mixture of 10% sodium nitroprusside, 10% $K_3Fe(CN)_6$, 0·9 *N* NaOH and water (1 + 1 + 1 + 15) can be used instead of **I**. K. Saito

3378. **Micro-determination of 2-aminothiophenol.** E. Ružička (Chem. Inst. High-School Pedag., Olomouc, Czechoslovakia). *Chem. Listy*, 1957, **51** (5), 969–973.—By the reaction with 2-aminothiophenol (**I**) in slightly acid ethanolic soln., ethoxyresazurin (**II**) is quant. reduced to the corresponding leuco compound (colour change, red to greenish blue). This reaction can be used for volumetric purposes. It is better to carry out the reaction in 4 *N* HCl, in which **II** is more sol., being converted into ethoxyresorufin. *Procedure*—Warm a known vol. of 0·001 *M* **II** in 4 *N* HCl to 80° and titrate with about 0·001 *M* **I** till a bluish-green colour appears (1 ml of 0·001 *M* **II** ≡ 0·2500 mg of **I**). J. ZÝKA

3379. **Ring analysis of mineral oil fractions by means of nomograms, using kinematic viscosity, refractive index and density.** J. Cornelissen and H. I. Waterman (Univ. Chem. Eng., Delft, Holland). *J. Inst. Petrol.*, 1957, **43**, 47–56.—Nomograms are constructed and used to enable the average no. of rings per mol. present of aromatic and naphthenic structures in mineral oil fractions to be determined from η, n and d. S.C.I. ABSTR.

3380. **Determination of tetraethyl-lead in petrol.** J. Krotký (Lab. Výzkumného a Zkušebního Leteckého Ústavu, Letňany, Czechoslovakia). *Paliva*, 1956, **36**, 124–127.—Several methods for the determination of Pb in petrol were compared. (*i*) The Pb was pptd. as $PbBr_2$ with a soln. of Br in CCl_4, converted into $PbCl_2$ and determined polarographically. Alternatively, the Pb was extracted according to Inst. of Petroleum standard IP 116/47 and determined polarographically. (*ii*) An indirect method was based on treating $Pb(NO_3)_2$ with a known vol. of 0·1 *N* $K_2Cr_2O_7$ and determining the excess of $K_2Cr_2O_7$ colorimetrically. The excess of $K_2Cr_2O_7$ was also determined iodimetrically. (*iii*) The Pb was determined as sulphate, either directly or after pptn. as $PbBr_2$. All methods gave reproducible results. N. E.

3381. **The analysis of combustion products. V. The use of infra-red spectrometry for following high-temperature kinetics.** R. Burt and G. J. Minkoff (Imperial Coll. of Sci. and Technol., London, England). *Anal. Chim. Acta*, 1957, **16** (3), 259–268.—A technique is described for determining the progressive formation of gaseous combustion products *in situ* without interrupting the reaction. A double-beam i.r. spectrometer is used and the reaction cell is constructed with sapphire end-plates. The method is designed for reactions at 250° to 430°, and an experimental example is concerned with the determination of CO during the slow oxidation of propane at 411°. W. C. JOHNSON

3382. **Methods of analysis for functional groups in earth (mineral) waxes.** A. Sundgren and V. T. Rauhala (State Inst. for Tech. Res., Helsinki). *J. Amer. Oil Chem. Soc.*, 1957, **34** (1), 7–9.—Available methods for determining the acid value, sap. value, hydroxyl value and carbonyl value of peat waxes have been examined. The reliability of the results is usually over-estimated. For the determination of acid value, potentiometric titration to pH 9 is more accurate than direct titration with a colour indicator. For the determination of sap. value, the potentiometric method is preferred to direct titration. In the potentiometric method, after saponification the solution is adjusted to pH 3 by addition of ethanolic H_2SO_4. Then the sample is titrated with 0·5 *N* ethanolic KOH to two inflection points, one being the end-point of strong acid (H_2SO_4) and the other the end-point of the liberated high-molecular-wt. organic acids. The sap. value is calculated from the KOH used between these two points. For hydroxyl value, a very slightly modified method of Freed and Wynne is suitable; and for carbonyl value, a modified method of Findley and Brown (*Ibid.*, 1953, **30**, 291) is used. The saponification reaction is a balanced one and the equilibrium depends on the experimental conditions, *e.g.*, wt. of sample and time of saponification. G. HELMS

3383. **The quantitative analysis of Phenidone - hydroquinone [quinol] developers.** A. J. Axford. *Brit. J. Photogr.*, 1956, **103**, 88–89.—Pyrazolid-3-ones are quant. oxidised by iodine in strongly acid soln. to the corresponding pyrazolone. Quinol is not oxidised. To determine Phenidone (1-phenylpyrazolid-3-one) in a developer containing quinol, acidify 100 ml of soln. with H_2SO_4 to pH 0·5, boil for 15 min. to discharge SO_2, cool to 20° and add starch soln. Add a slight excess of 0·05 *N* iodine determined from a preliminary titration, set aside for 5 min., then back-titrate with 0·05 *N* $Na_2S_2O_3$. CHEM. ABSTR.

3384. **Determination of acetyl content of cellulose acetate by near infra-red spectroscopy.** J. A. Mitchell, C. D. Bockman, jun., and A. V. Lee (Tennessee Eastman Co., Kingsport, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 499–502.—The absorption due to the residual hydroxyl groups is measured at 1445 mμ at which wavelength there is a reasonably linear relationship between concn. of acetyl and absorption. Pyrrole is a suitable solvent. The analysis of five commercial samples with acetyl concn. from 37·5 to 43·5% showed a standard deviation of ±0·22%, and there was close agreement with methods involving saponification or carbanilation. G. P. COOK

3385. **Determination of rosin acids in admixture with fatty acids.** A. Linder and V. Persson (Linder Patents, Stockholm). *J. Amer. Oil Chem. Soc.*, 1957, **34** (1), 24–27.—The authors describe improvements in their previously reported method (*Svensk Papperstidn.*, 1949, **52**, 331) for estimating rosin acids in the presence of fatty acids, by esterification of the fatty acids with azeotropic distillation of the water formed. Benzenesulphonic acid, previously used to retard rosin acid esterification, is replaced by H_2SO_4 whereby the esterification time is reduced from between 1 and 1·5 hr. to 20 min. Also, a packed column is introduced between the reaction flask and the condenser for water separation, in order to increase the concn. of benzene in the vapours. The esterification soln. consists of *n*-butanol (500 ml), benzene (500 ml) and conc. H_2SO_4 (6 g) refluxed for 30 min. to form butyl hydrogen sulphate. Other procedures are as before. The apparatus is illustrated, and comparative figures with those obtained by the Herrlinger - Compeau method (*Ibid.*, 1952, **29**, 342) are given. Light coloured acids are titrated to phenolphthalein and dark products, *e.g.*, tall oil, potentiometrically. G. HELMS

3386. **Titration of basic groups in ion-exchange resins with perchloric acid in glacial acetic acid.** G. E. Ficken and E. S. Lane (A.E.R.E., Harwell,

England). *Anal. Chim. Acta*, 1957, **16** (3), 207–209.—*Procedure* (*i*)—Boil a sample of resin containing 0·3 to 0·4 milli-equiv. of titratable N with 20 ml of acetic acid for 1 min. This treatment swells and degrades the resin. Cool, and titrate with 0·1 *N* $HClO_4$ in acetic acid with the use of crystal violet or Oracet blue B as indicator. Conclude the titration with the mixture kept at $\simeq 100°$ until the end-point is stable for 1 hr. *Procedure* (*ii*)—Mix the sample with 15 ml of 0·033 *N* $HClO_4$ in acetic acid and heat in an oven at 100° for 24 hr. Filter off the resin, wash with 35 ml of hot acetic acid and titrate the total filtrate with 0·1 *N* Na acetate in acetic acid. Of these procedures the first yields results more nearly in agreement with those from micro-Dumas determinations of N.

W. C. JOHNSON

3387. **Analytical studies on the ageing of *iso*-cyanate-based [polymer] foams.** J. K. Buxbaum (E. I. du Pont de Nemours & Co., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 492–496.—The chemical course of the ageing reaction was followed by examining a series of commercial *iso*cyanate-based resilient foams, most of which had shown marked changes in physical properties as the result of exposure to ambient conditions. The ageing appears to be due to hydrolysis, which results in the formation of CO_2 and of solvent-soluble polymer fragments and an insoluble residue, which are similar in chemical structure to the original foam. Amine end-groups are also formed and can be detected in the foam by colour tests with bromophenol blue and in extracts of the soluble polymer fragments by colour formation with *p*-chlorobenzenediazonium chloride. Artificial ageing by exposure to 100% r.h. and 70° or 90° showed the formation of the same general type of products as the naturally aged samples.

G. P. COOK

3388. **Analysis of polyisoprenes by infra-red spectroscopy.** J. L. Binder and H. C. Ransaw (Firestone Tire and Rubber Co., Akron, Ohio, U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 503–508.—The amounts of *cis*-1:4, *trans*-1:4, 1:2 and 3:4 additions in polyisoprenes can be determined by measuring the absorbances at 8·84, 8·68, 10·98 and 11·25 μ, respectively. For emulsion, bulk, sodium and potassium polyisoprenes, a modified base-line method must be used to determine the *cis*-1:4 and *trans*-1:4 absorbances. K. A. PROCTOR

3389. **Complexometric determination of total sulphur in synthetic elastomers containing chlorine and nitrogen.** B. B. Bauminger (Central Res. Div., Dunlop Res. Centre, Birmingham). *I.R.I. Trans.*, 1956, **32**, 218–233.—The S is converted by combustion into oxides, absorbed in H_2O_2 and pptd. as $BaSO_4$ by a known addition of $BaCl_2$. The excess of $BaCl_2$, and hence the content of S of the sample, is estimated in the presence of $BaSO_4$ by adding zinc disodium ethylenediaminetetra-acetate and Eriochrome black T and titrating with EDTA (disodium salt) until the colour changes from wine-red to blue. Small amounts of N, H_2O_2 and $ZnCl_2$ do not interfere. The order of accuracy is equal to that of the slower gravimetric technique.

F. J. M.

See also Abstracts 3221, 3282, 3284, 3470, 3504.

4.—BIOCHEMISTRY

INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

3390. **Refractometric determination of total solids and water of serum and urine.** M. E. Rubini and A. V. Wolf (Inst. Res., Walter Reed Army Med. Center, Washington, D.C., U.S.A.). *J. Biol. Chem.*, 1957, **225** (2), 869–876.—The accurate, precise and rapid method described is particularly useful for the determination of total solids in urine. The determination of total protein is not adversely affected by changes in the albumin to globulin ratio, and the usual lipid content of serum does not affect the determination. Serum and urine can be stored for longer than two months at −18° and then thawed without any significant change in refractive index, and they can be kept in an ordinary refrigerator for at least two weeks. J. N. ASHLEY

3391. **Direct measurement of the oxygen saturation of human blood during cardiac catheterisation.** G. A. Mook and W. G. Zijlstra. *Proc. Kon. Ned. Akad. Wet., C*, 1957, **60** (2), 158–166 (in English).—A special type of cuvette connected to the catheter, incorporating a magnetic stirring device, is described. This cuvette is used in conjunction with a "cyclops" reflection oximeter for instantaneous measurement of the oxygen saturation of the circulating blood. H. F. W. KIRKPATRICK

3392. **Procedure for serum-potassium determination. I. An improvement in cobalt colorimetry.** R. F. L. Maruna (Eijkman Inst., Djakarta, Indonesia). *Clin. Chim. Acta*, 1957, **2** (1), 21–24.—The qualitative test for Co by means of alkali thiocyanate in acetone has been examined, and directions are given for its use as a quantitative method for the range 0 to 100 mg-% of Co in H_2SO_4 medium.

N. E.

3393. **Colorimetric micro-determination of magnesium.** J. Carles (Catholic Inst., Toulouse, France). *Bull. Soc. Chim. Biol.*, 1957, **39** (4), 445–451.—To a sample soln. (6 ml) containing > 3 μg of Mg per ml add 0·1% gelatin soln. (1 ml), saturated aq. $CaSO_4$ soln. (1 ml) and 0·05% aq. Titan yellow soln. (1 ml). Shake well and add 5 *N* NaOH (1 ml). After 10 min. read the extinction at 540 mμ against a water blank treated similarly. Beer's law is followed with amounts of Mg up to 3 μg per ml of test soln. and a standard curve is not required.

H. F. W. KIRKPATRICK

3394. **Improvements in or relating to urine calcium test.** H. Sulkowitch. Brit. Pat. 769,289; date appl. 31.5.55. U.S.A., date appl. 9.6.54.—A reagent for detecting Ca in body fluids comprises a mixture having the following preferred composition—oxalic acid (40 g) and ammonium oxalate (25 g), with K H phthalate (50 g), KH_2PO_4 (35 g) and $KHCO_3$ (24 g) added as buffer, and chlorophenol red (0·25 g) as indicator. This mixture, which has a pH of $\simeq 3{\cdot}5$, is made up into 0·5-g tablets. To make the test, one tablet is dropped into a test-tube containing 1 to 10 ($\simeq 5$) ml of urine. A fine white ppt. indicates a normal content of Ca, no ppt. a deficiency of Ca, and a milky ppt. an excess of Ca.

J. M. JACOBS

3395. **Flame-photometric determination of calcium in biological material. Effect of low-level impurities from calcium oxalate precipitation.** T. Y. Toribara, P. A. Dewey and H. Warner (Dept. of Radiation Biology, Univ., Rochester, N.Y., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 540–543.—A routine procedure is described for the determination of Ca in serum by flame-photometric measurement after pptn. of the Ca as oxalate from a deproteinised solution. The flame emission is affected by small amounts of ammonium salts which might be carried from the separation procedure and by the type of acid used to dissolve the oxalate. The solubility of Ca oxalate accounts for small losses of Ca. By standardising the procedure, all the variables can be combined into a single correction factor. The method gave results all within 2% of the true values. K. A. PROCTOR

3396. **An enzymic estimation of D(+)-glucose in blood.** G. Pfleiderer and L. Grein (Inst. für Organ. Chemie, Univ. Frankfurt a.M.). *Biochem. Z.*, 1957, **328** (7), 499–506.—The following technique has been adopted for routine clinical use. In a 4-ml cuvette (thickness 1 cm) are placed the following—0·04 ml of phospho-enol-pyruvic acid ($1{\cdot}22 \times 10^{-6}$ *M*), 0·03 ml of reduced diphosphopyridine nucleotide ($3{\cdot}6 \times 10^{-7}$ *M*), 0·1 ml of KCl soln. (2 *M*), 0·1 ml of $MgSO_4$ soln. (0·5 *M*), 0·1 ml of adenosine triphosphate ($8{\cdot}5 \times 10^{-6}$ *M*), 0·01 ml of lactic acid dehydrogenase (50 μg of protein), 0·01 ml of pyruvate kinase (50 μg of protein), 0·05 ml of hexokinase (100 μg of protein) and 2·45 ml of triethanolamine buffer (0·05 *M*) (pH 7·6). The initial extinction at 340 mμ is determined after 5 min. and the reaction is started by adding 0·1 ml of the protein-free fluid obtained from the blood sample (≡0·01 ml of blood). A linear relationship between extinction and glucose concn. over the range 0 to 35 μg per 0·1 ml of test soln. is illustrated. Estimates in normal and in diabetic blood by this method give values that are about 20 to 25 mg per 100 ml lower than those determined by the Hagedorn and Jensen method. The enzymic method is claimed to have an accuracy within ±3%. G. W. CAMBRIDGE

3397. **Quantitative determination of indican in urine.** W. Feldheim (Anstalt f. Vitaminforsch. und Vitaminprüf., Potsdam-Rehbrücke). *Pharmazie*, 1957, **12** (2), 82–84.—The method of Obermayer and Jolles for the determination of indican in urine has been studied, with special attention to the time and temp. for development of colour. *Procedure*—Dilute the sample (20 ml) with H_2O to 200 ml. Shake a 4-ml aliquot with 20% aq. trichloroacetic acid (5 ml), 5% ethanolic thymol soln. (0·5 ml) and Obermayer's reagent (8 ml). Heat at 70° for 15 or 20 min., cool and extract with $CHCl_3$ (3 × 10 ml). Evaporate the combined extracts almost to dryness, cool and dilute with $CHCl_3$ to 4 ml. Determine the extinction in 1-cm cells with a Pulfrich photometer and filter S57. A. R. ROGERS

3398. **Determination of free and combined procaine in urine.** N. B. Krŷnskaya (Naval Medical Acad., Second Military Surgery). *Lab. Delo*, 1957, **3** (2), 27–29.—Procaine is determined by diazotisation of the amino group followed by coupling with thymol and measurement of the extinction at 478 mμ. To determine free procaine in urine, the sample (1 ml) is mixed with 0·5 ml of 9% HCl soln. and treated with 1 ml of freshly prepared 0·1% $NaNO_2$ soln., and then set aside for 5 min., after which 5 ml of a freshly prepared 1% soln. of thymol in *N* NaOH is added and the soln. is examined in a photometer which has been calibrated against a standard soln. To determine total procaine, the sample (1 ml) and 1 ml of 9% HCl soln. are heated on a boiling-water bath for 30 min., and the hydrolysed product is treated with $NaNO_2$ soln., etc., as above. G. S. SMITH

3399. **Method for the quantitative estimation of 1-*cyclo*hexylamino-2-propyl benzoate (Cyclaine) [hexylcaine] and derived products in biological tissues.** V. D. Wiebelhaus and E. R. Wynosky (Merck Inst. Therapeutic Res., West Point, Pa., U.S.A.). *J. Lab. Clin. Med.*, 1957, **49** (4), 651–656.—A scheme is described for the separate determination of hexylcaine (**I**), of the amino alcohol (**II**) formed by hydrolysis of **I**, and of the amide (**III**) produced by the alkaline or enzymatic rearrangement of **I**. After making alkaline, all three compounds are extracted from biological fluids into 1:2-dichloroethane. On shaking the extract with 0·2 *N* HCl, only **III** remains in the organic phase. One portion of the acid extract is examined directly for free **II**, and the other portion for total **II** after alkaline hydrolysis of the **I**. The 1:2-dichloroethane containing the **III** is similarly hydrolysed to **II** for the colorimetric determination, which is based on the formation of a solvent-soluble complex with the sodium salt of methyl orange. The complex is extracted into 1:2-dichloroethane, the excess of methyl orange is removed by centrifugation, and the extinction of the solvent phase is determined after acidification with ethanolic H_2SO_4 at 540 mμ. Neither **I** nor **III** can be determined directly by the method. Results are given on plasma and urinary levels attained after administration of **I** and these are compared with those after administration of procaine. W. H. C. SHAW

3400. **The role of nitrite concentration in the estimation of phenazone [in plasma].** G. A. Zak (New York Univ. Coll. Medicine, N.Y., U.S.A.). *J. Lab. Clin. Med.*, 1957, **49** (4), 623–625.—Improved results are obtained in the determination of phenazone in plasma by the method of Brodie *et al.* (*Brit. Abstr. C*, 1949, 342) by closer control of the $NaNO_2$ concn. *Procedure*—To 1 ml of plasma filtrate add 50 μl each of 1·3 *N* H_2SO_4 and of 0·1% $NaNO_2$ soln. Measure the extinction in a 1-cm cell at 350 mμ between 23 and 28 min. after the addition of the reagents. W. H. C. SHAW

3401. **Detection of hog bile in mixtures with ox or sheep bile.** P. Jacobs (N.V. Fabriek van Chemische Producten, Vondelingenplaat, Holland). *Pharm. Weekbl.*, 1957, **92** (8), 239–246.—A simple, reliable and selective method is described which can be applied to fresh bile, to bile which has been stored for various periods of time and preserved with different agents, and to inspissated bile, bile extract and bile powder. Cryst. NaCl (4 to 6 g) is added to about 10 ml of bile in a test-tube and shaken vigorously for a short time. A heavy resinous or jelly-like ppt., usually in big, sticky lumps, reveals the presence of at least 25% of hog bile. Some simple refinements make it possible to detect hog bile in quantities as small as 2 to 5%. P. HAAS

3402. **Separation of purines and pyrimidines by paper electrophoresis.** G. Semenza (Inst. of Biochem., Uppsala Univ., Sweden). *Acta Chem. Scand.*, 1957, **11** (1), 24–27 (in English).—For the

quant. determination of purines and pyrimidines, paper electrophoresis is more rapid and accurate than paper chromatography. Separation of the bases from a nucleic acid hydrolysate can be performed only at acid pH; at neutral or alkali pH, the low solubility of adenine and guanine causes tailing. The order of travelling towards the cathode at pH 3·8 is cytosine (**I**), adenine (**II**), thymine plus uracil (**III**), guanine (**IV**); at pH 3 it is **I**, **II**, **IV**, **III**. A pH of 3 was chosen; at this pH, the relative mobilities agree fairly well with those calculated from dissociation constants. A hydrolysate of deoxyribonucleic acid (from calf thymus) was taken up in enough *N* HCl to contain 5 to 50 μg of each base in 5 μl. A humid chamber, horizontal electrophoresis apparatus, with Whatman No. 1 paper strips and citrate - HCl buffer, pH 3, diluted to an ionic strength of 0·02, was used. Samples of 5 μl were applied, part of the paper being left free for blanks. A voltage of 440 V, 7 to 8 mA, was applied for 3 hr. at room temp. After drying the paper for 5 min. at 100°, compact spots were located. They were eluted overnight with 3·5 ml of 0·1 *N* HCl, and readings were taken with a Beckman DU spectrophotometer against water at 250, 260, 280 and 290 mμ. W. J. Wright

3403. **Rapid determination of serum lipids. II. Fatty acid determination by a formaldehyde reaction.** W. Weigel (II Med. Univ.-Klinik der Charité, Berlin). *Fette, Seif., Anstrichmitt.*, 1956, **58** (12), 1038–1041.—In the presence of conc. H_2SO_4, fatty acids react with formaldehyde to give a yellow to red - brown polymerisation product which can be determined absorptiometrically. For determinations on blood serum, the fat is extracted by the method of Bloor, cholesterol and pigments are removed, and the unsaponifiable matter (from 0·33 ml of serum) is taken up in conc. H_2SO_4 (3 ml) and a 10% soln. of formaldehyde (0·5 ml). The mixture is maintained at 98° to 100° for 60 min., the soln. is then cooled and diluted with conc. H_2SO_4 (3 ml) and the colour is measured in a Pulfrich photometer with S430 filter. A blank experiment is carried out at the same time. The total fatty acid content is then read from a calibration curve prepared with stearic acid. If the reaction is carried out at lower temp., only the unsaturated acids react. These acids can also be determined by the following reagents under the conditions given—(*i*) a mixture of H_2SO_4 and acetic anhydride (3 + 1), 60 min. at 100°; (*ii*) 10% formaldehyde soln. (0·5 ml), 3 ml of H_2SO_4 plus 1 ml of H_2O, 60 min. at 88° to 89°; (*iii*) 0·5 ml of 4% dimethylaminobenzaldehyde in 50% H_2SO_4, 20 min. at 88°. (*Cf. Anal. Abstr.*, 1956, **3**, 804.) E. Hayes

3404. **Improvements in the technique of estimating amino acids (and other substances) by paper-sheet chromatographic methods.** J. W. H. Lugg and E. McEvoy-Bowe (Dept. Biochem., Univ. of Malaya, Singapore). *Nature*, 1957, **179**, 1076.—To improve existing procedures for the estimation of amino acids separated on paper chromatograms the following conditions must be standardised and closely controlled—(*i*) the size of the original spot of liquid, (*ii*) the compositions of the mobile and stationary liquid phases, (*iii*) the temp. of operation, (*iv*) the extent of traverse of the mobile phases, (*v*) the basicity and solvent properties of the colour-developing soln., (*vi*) the humidity, temp., and time for which the treated papers are heated. It is desirable also to use a circular, small-orifice type of densitometer for measuring the light absorption of the developed bands, and to bleach the band and re-measure the light absorption after re-orientating the paper in the densitometer in the position formerly yielding max. absorption.

K. A. Proctor

3405. **Studies in histochemistry. XLV. Determination of hydroxyproline in microgram amounts of tissue.** B. W. Grunbaum and D. Glick (Minnesota Univ. Med. Sch., Minneapolis, U.S.A.). *Arch. Biochem. Biophys.*, 1956, **65** (1), 260–267.—The method is based on the colour given by hydroxyproline after oxidation with alkaline H_2O_2 in the presence of $CuSO_4$ with *p*-dimethylaminobenzaldehyde, and is a modification of that described by Neuman and Logan (*J. Biol. Chem.*, 1950, **184**, 299). The concn. of reagents required has been re-investigated and manipulative improvements made so that amounts of hydroxyproline down to 2·5 mμg may be determined in tissue samples, such as microtome sections. Interference by tyrosine under the improved conditions is negligible. A simplified procedure for the preliminary hydrolysis of samples requires heating of the sample (5 to 50 μg) with 20 μl of 6 *N* HCl at 170° for 1 hr. in a sealed tube. After cooling, the tube is opened and the contents dried *in vacuo* over NaOH pellets.

W. H. C. Shaw

3406. **A new technique for the analysis of histidine, tyrosine, methionine and arginine in protein hydrolysates.** H. Fraenkel-Conrat and B. Singer (Virus Lab., California Univ., Berkeley, U.S.A.). *Arch. Biochem. Biophys.*, 1956, **65** (1), 296–301.—Aliquots of hydrolysates from 2 to 5 mg of protein sample containing 0·01 to 0·2 μmole of histidine, arginine and tyrosine and 0·1 to 1 μmole of methionine are chromatographed on Whatman No. 1 paper, and developed with the upper phase of butanol - acetic acid - water (4 : 1 : 5). After location of the amino acids with spray reagents, the corresponding bands from unsprayed chromatograms are cut out and individual amino acids determined colorimetrically by the four procedures given. Recovery of the amino acids added to hydrolysates is quant. The method is used for the examination of certain crystalline proteins and virus preparations, and the significance of the results is discussed.

W. H. C. Shaw

3407. **The anion-exchange resins Dowex 1 and 2. Immediate separation of organic and inorganic iodine. Rapid estimation of iodinated amino acids labelled with ^{131}I.** P. Blanquet, G. Meyniel, J. Mounier and C. A. Tobias (Fac. of Med. and Pharm., Bordeaux, France). *Bull. Soc. Chim. Biol.*, 1957, **39** (4), 419–424.—The resins are treated with 2 *N* NaOH and washed copiously with water. Columns 15 cm long and 1·5 cm in diameter are prepared and the soln. (*e.g.*, thyroid hydrolysate) is passed through. Elution is effected with 0·1 or 0·2 *N* HCl and the eluate is collected in 1-ml fractions at a rate of 40 to 50 ml per hr. Thyroxine, monoiodotyrosine and di-iodotyrosine appear in the eluate in that order and are estimated by counting the radioactivity; iodide remains on the column. Dowex 1 yields a rapid separation of the amino acids; with Dowex 2 the separation takes much longer, but is more complete.

H. F. W. Kirkpatrick

3408. **Application of high-tension electrophoresis to the total quantitative analysis of protein hydrolysates. Investigation of lysozyme.** K. Dose and A. Caputo (Max Planck Inst. für Biophysik, Frankfurt am Main). *Biochem. Z.*, 1956, **328** (5), 376–385. —The separation, by high-tension electrophoresis, of the amino acids in hydrolysates of Lysozym (Armour) (either crystalline or after exposure to X-rays) is described. The technique used was that of Wieland and Fischer (*Naturwissenschaften*, 1948, **35**, 29), and details are given of the pH, voltage gradient and duration of electrophoresis required in each of the six stages. Adequate separation of most of the amino acids was achieved by this means, but serine, threonine, methionine, valine, leucine and *iso*leucine were separated from one of the fractions by chromatography. The individual amino acids were estimated by spraying with ninhydrin (0·5% soln. in *n*-butanol - pyridine) (100:5), drying for 30 min. at 90°, extracting with methanol and treating with methanolic Cu acetate soln. The colour was then measured in a spectrophotometer at 510 mμ. The values for the amino-acid content of the hydrolysates so obtained are compared with those obtained with microbiological methods. G. W. CAMBRIDGE

3409. **Determination of microgram quantities of protein in biological fluids. The estimation of plasma and serum protein in spinal fluid.** G. R. Kingsley and G. Getchell (Dept. of Physiol. Chem., Univ. of California, Los Angeles, U.S.A.). *J. Biol. Chem.*, 1957, **225** (1), 545–556.—A simple, accurate colorimetric method is described for the determination of the total protein in spinal fluid and serum. It is based on the formation of a blue colour in the presence of tetrabromophenolphthalein ethyl ester in weakly acid soln., the colour being measured photometrically at 600 mμ. The method, which is sensitive to 0·1 μg of protein, is unsatisfactory for urinary proteins, but is applicable to the determination of serum and plasma proteins in the presence of spinal fluid. J. N. ASHLEY

3410. **Determination of serum cholesterol.** A. A. Henly (Biochem. Lab., Little Bromwich Gen. Hosp., Birmingham, England). *Analyst*, 1957, **82**, 286–287.—The method described is a modification of that of Zlatkis *et al.* (*J. Lab. Clin. Med.*, 1953, **41**, 486). The reagent is a 0·05% soln. of $FeCl_3.6H_2O$ in pure acetic acid (purification described). The serum or plasma (0·1 ml) is added to 10 ml of the reagent and, after the proteins have flocculated, the liquid is centrifuged. The clear liquid (5 ml) is transferred to a glass-stoppered test-tube. Similar tubes contain 5 ml of the reagent (the blank) and 5 ml of a standard cholesterol soln. (0·2 mg in the reagent), respectively. To each tube 3 ml of H_2SO_4 are added and the contents are thoroughly mixed. After an interval for full colour development (≃ 30 min.) the purple colours are measured, either in an E.E.L. colorimeter with an Ilford No. 626 filter or in a spectrophotometer at 560 mμ. The method is convenient for determining free and ester cholesterol (both give equiv. colours with the reagent) after their chromatographic separation. A. O. JONES

3411. **Method for determining total blood cholesterol.** D. Giarnieri (Ist. di Chim. Biol., Univ. di Camerino, Italy). *Ric. Sci.*, 1957, **27** (2), 389–391. —The method described consists in precipitation of the serum proteins, extraction of the total cholesterol by ethanol - ether, and determining the cholesterol colorimetrically by the Liebermann - Burchard reaction. *Procedure*—Serum (0·80 ml) is treated dropwise with 19·20·ml of a mixture of equal vol. of ethanol and ether in a stoppered flask and set aside for 90 min. The mixture is then filtered through a rapid filter-paper and 10 ml of the filtrate (equiv. to 0·4 ml of serum) is carefully evaporated to dryness over a water bath at 40° to 55°. The residue is taken up with 10 ml of $CHCl_3$ and placed, together with a previously prepared sample of Liebermann's reagent, in a thermostat at 37°. After 10 min., 4 ml of the reagent is added to the $CHCl_3$ soln. and the mixture is once more held at 37° for 15 min. The colour is then compared at 620 mμ with that of a freshly prepared control (a mixture of 10 ml of $CHCl_3$ and 4 ml of acetic anhydride). The reading is referred to a standard curve. Details are given of a method for giving increased accuracy to the determination. P. HAAS

3412. **Estimation of the neutral and phenolic ketosteroids in urine with pyridine and *m*-dinitrobenzene.** W. Wendland and K. Lohmann (Inst. für Medizin und Biologie, Dtsch. Akad. der Wissenschaften, Berlin). *Biochem. Z.*, 1957, **328** (6), 420–428.—The reaction of 17-oxosteroids with dinitrobenzene in alkaline soln. has been investigated with a view to eliminating the excessive blank reaction and interference by other compounds such as creatinine which are drawbacks to the Zimmermann reaction for the estimation of 17-oxosteroids in urine. It has been found that the use of pyridine as a solvent for the dinitrobenzene and sodium hydroxide, instead of alcohol as used in the original method, overcomes these difficulties. In addition, the method is claimed to be six to seven times as sensitive, and the intensity of the "hormone colour" is directly proportional to the hormone concentration. It is essential that the pyridine, toluene and dinitrobenzene used should be specially purified, and details are given. Two methods are described for the estimation of neutral ketosteroids and the neutral and phenolic ketosteroids in urine. G. W. CAMBRIDGE

3413. **The analysis of urinary 17-oxosteroids by gradient elution.** A. E. Kellie and A. P. Wade (Courtauld Inst. of Biochem., Middlesex Hosp. Med. Sch., London). *Biochem. J.*, 1957, **66** (2), 196–206.—A new method is described for the analysis of the 17-oxosteroid fraction of urine. It avoids formation of artifacts during hydrolysis of conjugates and gives improved separation of individual 17-oxosteroids. Hydrolysis of the 17-oxosteroids is effected with limpet (*Patella vulgata*) β-glucuronidase and, subsequently, that of 17-oxosteroid sulphates by continuous extraction of an acidified soln. with ether. The individual 17-oxosteroids are separated chromatographically by gradient elution; there is an improved separation of the 11-oxygenated 17-oxosteroids present in normal urine. J. N. ASHLEY

3414. **Colorimetric micro-determination of corticoids by use of 4:7-diphenyl-1:10-phenanthroline.** N. M. Mazarella (Metabolic Res. Lab., Veterans Admin. Hosp., Washington, D.C., U.S.A.). *J. Biol. Chem.*, 1957, **225** (1), 239–246.—The ferroin reaction is used in the two simple methods described. In the first method 2:2′-dipyridyl is the reagent and can be used for the determination of reducing steroids in amounts greater than 25 μg. In the second method bathophenanthroline (4:7-diphenyl-

1:10-phenanthroline) is used for micro-determinations when max. sensitivity is required. This reagent forms a ferrous complex with max. absorption at 533 mμ and mol. extinction coeff. of 22,400. Esters of the various corticosteroids cannot be determined by these methods or used as standards. The reactions are given by steroids with a free α-ketol grouping at $C_{(20)}$ and $C_{(21)}$. J. N. ASHLEY

3415. **The identification of corticosterone in human plasma and its assay by isotope dilution.** R. E. Peterson (Nat. Inst. of Health, Bethesda, Md., U.S.A.). *J. Biol. Chem.*, 1957, **225** (1), 25–37.—An isotope dilution method with [4-^{14}C]corticosterone is used for the determination of corticosterone in plasma. The method is useful because the plasma extract can be considerably purified without concern about minor losses of steroid. Because of this purification and the use of a fluorimetric method of assay on the relatively pure product, a considerable degree of specificity and sensitivity is attained. J. N. ASHLEY

3416. **Spectrophotometric method for the analysis of binary mixtures of urinary steroids.** J. Bitman, J.-P. Rosselet, A. de M. Reddy and S. Lieberman (Dept. of Biochem., Columbia Univ., New York, U.S.A.). *J. Biol. Chem.*, 1957, **225** (1), 39–52.—The method described is used for the analysis of binary mixtures of saturated and unsaturated urinary steroids. It is based on the differences in spectra of these steroids in H_2SO_4. The concn. of each steroid in the following chromatographically homogeneous pairs can be determined: *epi*androsterone and dehydro*epi*androsterone, androsterone and Δ⁹-androstenolone, and aetiocholanolone and Δ⁹-aetiocholenolone. With chromatographic fractions from urinary extracts the method gives results that agree with those obtained by a chemical method which involves isolation of the unsaturated compounds as their epoxides. J. N. ASHLEY

3417. **Micro method for the determination of carbonic anhydrase.** E. Cutolo (Centro di Studio per Enzymologia, Rome, Italy). *Ric. Sci.*, 1957, **27** (2), 386–388.—A Whatman No. 1 filter-paper, previously sprayed with a 1 to 2% ethanolic soln. of thymol blue, is moistened at two points *A* and *B* with 5 μl of a 0·1 *N* soln. of $NaHCO_3$, and 5 μl of a soln. used for dilution of the enzyme (preferably a 0·05% soln. of peptone) is immediately delivered on to *A* and 5 μl of the enzyme soln. suitably diluted is placed on *B*. The surface of the paper so prepared gives an effect comparable with that produced by shaking a Warburg apparatus. The action of the enzyme sets free CO_2, with subsequent change in pH and in the indicator colour. Alternatively, Merck's universal indicator paper may be used, but the $NaHCO_3$ soln. must then be buffered to ≃ pH 7·5. As a standard of reference for judging the activity of an enzyme, a sample of carbonic anhydrase was prepared by Keilin and Mann's method (*Biochem. J.*, 1940, **34**, 1163). A relatively pure specimen of this dissolved in 5 μl of a soln. containing 30 μg of protein having a zinc content of 0·15%, equiv. to 30 specific units per ml (or 1000 enzyme units per mg, according to Keilin and Mann) and used as described above, gives a blue colour corresponding to a pH of about 10. For a blood test, 5 μl of blood is laked with 0·1 ml of water; 5 μl of such hæmolysed blood is used for testing the activity. For red blood cells, 1 ml of sediment from 5 μl of hæmolysed blood in 100 ml is used for testing. P. HAAS

3418. **Determination of phosphorus and phosphatase with N-phenyl-*p*-aminodiphenylamine [*p*-semidine].** R. L. Dryer, A. R. Tammes and J. I. Routh (Dept. Biochem., State Univ., Iowa City, U.S.A.). *J. Biol. Chem.*, 1957, **225** (1), 177–183.—*p*-Semidine is used as a reagent for the reduction of molybdophosphate. It is stable, the reduction is rapid, and the reagent contributes to the optical absorbance of the final soln. Max. absorbance is obtained in 10 min. or less and remains const. for at least 1·5 hr., and is determined spectrophotometrically at 345 to 350 mμ or 770 mμ. This new reagent is used for the determination of lipid P, serum inorg. P and serum alkaline phosphatase. J. N. ASHLEY

3419. **Convenient methods of titrating penicillinase and antipenicillinase serum.** S. Bŷrdarov (Microbiol Dept., Med. Faculty, Sophia, Bulgaria). *Antibiotiki*, 1957, **2** (2), 19–21.—To determine penicillinase, various dilutions of the sample soln. are mixed with the same amount of penicillin and kept at 37° for 1 hr. Drops from each of the soln. are placed on paper in contact with agar treated with the test organism *Staphylococcus aureus* 209. After 15 to 20 hr. at 37°, each paper is examined for the presence of a sterile zone. To determine antipenicillinase, various dilutions of the sample soln. are mixed with penicillinase and kept at 37° for 1 hr., after which penicillin is added and the soln. are again kept at 37° for 1 hr. The further procedure is as described above. G. S. SMITH

See also Abstracts 3314, 3435, 3439.

Drugs

3420. **The morphine - Marmé complex.** L. Levi (Dept. Nat. Health and Welfare, Ottawa, Canada). *Anal. Chem.*, 1957, **29** (4, Part I), 470–474.—A study of the composition of the complex formed between morphine and aq. CdI_2 or CdI_2 - KI (Marmé reagent) and of the mechanism of the reaction is presented. Hitherto unreported constants of the derivative, which may be used advantageously for detecting and characterising minute amounts of the alkaloid, are also recorded. The study included u.v., i.r., X-ray diffraction and optical rotation measurements. The sensitivity of the reaction is dependent primarily on the concn. of KI in the reagent and the ratio of reagent to reactant volume in the final system; when these conditions are carefully adjusted, as little as 0·1 μg of the alkaloid can be readily detected. G. P. COOK

3421. **Reactions of some synthetic analgesics compared with morphine and its derivatives.** M. Hädicke and M. Kuntze (Staatlichen Inst. f. Arzneimittelprüfung, Jena). *Pharm. Zentralh.*, 1957, **96** (4), 152–153.—Reactions with ammonium molybdophosphate reagent, HNO_3 - H_2SO_4 reagent and Marquis reagent are recorded for methadone, isomethadone, pethidine, morphine, dihydrohydroxycodeinone hydrochloride, codeine, ethylmorphine, dihydrocodeinone, dihydromorphinone and papaverine. A. R. ROGERS

3422. **Fluorimetric determination of 11-demethoxyreserpine.** J. A. Gordon and D. J. Campbell (Abbott Lab., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 488–490.—A study of the fluorimetric properties of the alkaloid showed a

single fluorescent max. at 360 mμ and two activating max. at 280 and 365 mμ. The intensity of the fluorescence is increased tenfold after reaction of the alkaloid with Ce^{IV} in 5 *N* acetic acid soln. containing 0·1 *N* H_2SO_4. A linear response is obtained up to 5 μg, and as little as 0·5 μg can be detected. The method was applied to the analysis of pharmaceutical mixtures. G. P. Cook

3423. **Colorimetric estimation of dihydrostreptomycin and streptomycin.** R. Natarajan and J. N. Tayal (T.D.E. Lab., Kanpur, India). *J. Pharm. Pharmacol.*, 1957, **9** (5), 326–329.—This rapid method is based on the red colour produced when streptomycin or dihydrostreptomycin is mixed with 1-naphthol and NaOBr in alkaline soln. *Reagent*—Dissolve NaOH (20 g) in H_2O (75 ml), cool, add Br (5 ml) and dilute with H_2O to 1 litre. *Procedure*—Mix an aq. soln. of the sample (containing ≃ 1 mg in 1 ml) with 10% NaOH (1 ml) and 0·02% ethanolic 1-naphthol (2 ml). After 15 min., add NaOBr reagent (1 ml) and CCl_4 (10 ml), shake well, add absolute ethanol (5 ml), shake again and measure the extinction of the aq. phase at 530 mμ in a 1-cm cuvette with a reagent blank. The results were in satisfactory agreement with those obtained by the B.P. microbiological method for six samples of dihydrostreptomycin sulphate. A. R. Rogers

3424. **Spectrophotometric determination of tyrothricin.** S. Z. Patel and J. S. Naravane (Alembic Chem. Works Co. Ltd., Baroda). *Indian J. Pharm.*, 1957, **19** (3), 67–69.—The chemical method of Rittenberg *et al.* (*J. Biol. Chem.*, 1947, **168**, 183) for the assay of tyrothricin (**I**) has been re-examined. Results obtained by the proposed modified method agree within ±2% with those by the hæmolytic method. *Procedure*—Evaporate an alcoholic soln. (containing ≃ 150 μg of **I**) almost to dryness, cool to room temp., add 5 ml of a 0·125% soln. of *p*-dimethylaminobenzaldehyde in 80% (w/v) H_2SO_4 to which 0·005% of $FeCl_3$ has been added, and shake thoroughly for 15 sec. After 15 min. add 0·2% aq. $NaNO_2$ (0·1 ml), shake for 15 sec., add H_2O (5 ml), mix thoroughly and allow to reach room temp. Compare the intensity of the blue colour at 620 mμ with that of a reagent blank.
A. R. Rogers

3425. **Turbidimetric assay of tetracycline, chlortetracycline, oxytetracycline, chloramphenicol, neomycin and polymyxin using a single test organism.** D. M. Wintermere, W. H. Eisenberg and A. Kirshbaum (Food Drug Admin., Washington, D.C., U.S.A.). *Antibiot. & Chemother.*, 1957, **7** (4), 189–192.—A general method for the named antibiotics with *Escherichia coli* (ATCC 10536) as test organism is described. The only modifications to the general method for individual antibiotics are in standard curve concn. and in the use of different diluent buffers. The procedure is not applicable to mixtures of the antibiotics unless preliminary separation by physical or chemical methods can be attained. Other common antibiotics do not interfere when present below the concn. listed.
W. H. C. Shaw

3426. **Analytical separation of insulin from whale pancreas by partition chromatography.** Yuichi Sasano and Katsumi Higashi (Tokyo Res. Lab., Taiyo Fisheries Co., Tsukishima, Chuo-ku, Tokyo). *J. Pharm. Soc. Japan*, 1956, **76** (12), 1359–1361.—In the preparation of pure insulin (**I**) from crude whale insulin by Porter's method (*Biochem. J.*, 1953, **53**, 320), with a mixture of acetic acid, butanol and water (1 + 3 + 4), **I** is always accompanied by protein impurity. In an investigation for a suitable developer for the separation, it was found that the optimum composition is 8% of acetic acid, 25 to 50% of butanol and 40 to 70% of water. K. Saito

3427. **Identification of barbiturates by microsublimation.** H. Rządkowska. *Acta Polon. Pharm.*, 1956, **13**, 57–63.—The feasibility of distinguishing the barbituric derivatives, phenobarbitone, barbitone, allobarbitone, methylphenobarbitone, hexobarbitone, cyclobarbitone and Irenal by microsublimation under normal conditions was investigated. The microsublimates had characteristic microscopical pictures, which differed in shape and grouping of individual crystals. By establishing the m.p. and optical appearance, the above compounds can be readily identified.
Chem. Abstr.

3428. **Analysis of crotylethylbarbituric acid. I. Detection and volumetric methods of determination.** W. Horsch (Pharm. Inst. Karl-Marx-Univ., Leipzig). *Pharmazie*, 1957, **12** (3), 122–125.—Paper-chromatographic methods of separation and colour and pptn. reactions are described for the identification of crotylethylbarbituric acid (vinbarbitone) (**I**). Cryst. derivatives include the *p*-nitrobenzyl ester (m.p. 133°) and the product of condensation with xanthhydrol (m.p. 223° to 224°). For the quant. determination of **I**, acidimetric titration in aq. or non-aq. soln., bromimetric titration or argentimetric titration can be used. The argentimetric method is recommended because it is relatively specific.

II. Volumetric determination in mixtures and pharmaceutical preparations. W. Horsch. *Ibid.*, 1957, **12** (4), 212–215.—To determine **I** in suppositories, titrate a soln. in dimethylformamide - benzene (3:1) with Na methoxide, with thymol blue as indicator. To determine **I** in the presence of caffeine, theobromine, phenazone, acetanilide, phenacetin, hydroxyethyltheophylline, codeine or amidopyrine, dissolve the sample in warm aq. ethanol and titrate with carbonate-free alkali, with thymolphthalein as indicator. To determine **I** in the presence of saturated barbiturates, allow the sample to react with excess of Br, and measure the excess by addition of KI and titration with $Na_2S_2O_3$. To determine **I** in the presence of acetylsalicylic acid, salicylamide, *N*-*iso*propylsalicylamide, phenacetin or caffeine, dissolve the sample in aq. Na_2CO_3 soln. and titrate with $AgNO_3$ soln. A. R. Rogers

3429. **Paper chromatography of some tuberculostatics.** Tsuyoshi Sunada (Dept. of Hyg., Osaka Univ. Med. Sch., Japan). *Med. J. Osaka Univ.*, 1956, **7** (3), 497–500.—R_F values are given for sodium and calcium *p*-aminosalicylate, isoniazid, pyrazinamide and dihydrostreptomycin sulphate by the use of the solvent systems butanol - conc. HCl (2:1), ethanol - conc. HCl (3:1) and butanol - ethanol - water (4:1:1). The colours developed by a wide range of spray reagents are also described. Complete separation of these tuberculostatic agents could be effected by two-dimensional paper chromatography and a combination of the above-mentioned solvent systems. The amount of each substance on the chromatogram can be determined by the usual methods. J. N. Ashley

3430. **Bromimetric determination of Veritol [pholedrine] and Paredrine (Pulsoton) [hydroxyamphetamine].** E. Varga and G. Vastagh (Staatlichen Inst. f. Hygiene, Budapest). *Pharm. Zentralh.*, 1957, **96** (4), 149–152.—This method for the determination of pholedrine and hydroxyamphetamine is based on quant. bromination in acid soln. *Procedure*—Dissolve the sample (≃ 15 mg of base) in 10% H_2SO_4 (5 ml), dilute with H_2O (10 ml) and add 0·1 *N* $KBrO_3$ (10·0 ml), KBr (0·5 g) and 50% H_2SO_4 (5 ml). Shake in a stoppered flask, add KI (0·5 g) and dilute with H_2O (50 ml). Titrate the iodine equiv. to the excess of Br with 0·1 *N* $Na_2S_2O_3$, with starch as indicator. Perform a blank titration. Recovery experiments indicate that the error is $< \pm 0{\cdot}5\%$. Leptazol does not interfere. A. R. ROGERS

3431. **Assay of menadione [menaphthone]—a polarographic procedure.** J. C. Jongkind, E. Buzza and S. H. Fox (Gelatin Products Div., R. P. Scherer Corp., Detroit, Mich., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (4), 214–216.—A simple method is given for the rapid determination of menaphthone (**I**) in complex pharmaceutical mixtures. It does not distinguish between **I** and 1:4-dihydroxy-2-methylnaphthalene. The coeff. of variation, based on 20 determinations, is 2·4%. *Procedure*—Shake the sample mechanically with benzene (20 ml) and a soln. of $(NH_4)_2Ce(NO_3)_6$ (0·65 g) in *N* H_2SO_4 (25 ml). Centrifuge and transfer an aliquot of the benzene layer (containing approx. 0·5 mg of **I**) to the electrolysis vessel. Dilute to 10 ml with benzene, add 0·2 *M* LiCl in anhydrous methanol (10 ml) and aq. NH_3 (0·1 ml), deoxygenate and record the polarogram. Add a standard soln. of **I** in benzene - methanol (1:1) (1 ml), deoxygenate and record the polarogram. Measure the diffusion currents and calculate the original concn. of **I** by difference. A. R. ROGERS

3432. **Colorimetric determination of pyrazine derivatives.** Saburo Kajiwara and Tsuyoshi Sunada (Dept. of Hyg., Osaka Univ. Med. Sch., Japan). *Med. J. Osaka Univ.*, 1956, **7** (3), 493–495.—Pyrazine derivatives such as pyrazinamide and pyrazinoic acid are determined spectrophotometrically at 500 mμ and 470 mμ, respectively, using sodium pentacyanoamminoferroate as the colorimetric reagent to give the orange - red complex, which is stable for longer than 24 hr. The method will determine 1 to 50 μg per ml of the amide and 1 to 45 μg per ml of the acid. When used as a spot test the limits of identification are 0·05 μg and 0·2 μg of amide and acid, respectively. J. N. ASHLEY

3433. **Identification of glucosulphone (Promin).** H. de Vries (Lembaga Pharmacotherapie, Djakarta, Indonesia). *Pharm. Weekbl.*, 1957, **92** (8), 237–238.—Additional data are provided on the identification of glucosulphone (*cf. Ibid.*, 1949, **84**, 348). (*i*) It is pptd. from soln. by methanol, and has no sharp m.p.; (*ii*) it reduces Fehling soln. rapidly on warming; (*iii*) $Ba(NO_3)_2$ and HNO_3 precipitate $BaSO_4$; (*iv*) it decolorises iodine soln. in acid or neutral medium; (*v*) it gives a positive reaction for a primary aromatic amine with 2-naphthol and $NaNO_2$; (*vi*) with phenylhydrazine it produces an impure product which is probably glucosazone. P. HAAS

3434. **Modification of the analysis of sulphonamides.** Kiichiro Kakemi, Toyozo Uno and Yoshihiko Ikegami (Pharm. Dept., Med. Fac., Kyoto Univ., Sakyo-ku, Kyoto). *J. Pharm. Soc. Japan*, 1956, **76** (11), 1331.—The end-point of the diazotisation of sulphonamides is easily detected by the use of a test paper impregnated with 3% dimethylaminobenzaldehyde. At the end-point the test paper fails to turn yellow on the addition of the reaction mixture. This paper appears to be superior to zinc iodide - starch paper. K. SAITO

3435. **Determination of 4-acetamido-N-lauroylnaphthalenesulphonamide.** Shigesi Toyoshima, Hiroshi Tatsumi and Miho Hashizume (Keio Univ., Tokyo). *Chemotherapy, Japan*, 1956, **4**, 22–26.—4-Acetamido-N-lauroylnaphthalenesulphonamide (**I**) cannot be assayed by the usual methods for sulpha drugs, such as with Tsuda reagent or with *p*-dimethylaminobenzaldehyde. The lauroyl group inhibits the formation of colour. The following procedure was established. **I** (0·5 to 3·5 mg) and 65% H_2SO_4 (4·0 ml) were heated for 1 hr., the reaction mixture was made slightly alkaline with NaOH, then made up to 50 ml and filtered. Ethanol (40 ml) and *N* HCl (3·0 ml) were added to the filtrate (2·0 ml) and the whole was shaken after adding diazotised *p*-nitroaniline soln. (1·0 ml) [prep. by adding 2·5% $NaNO_2$ soln. (0·15 ml) and then 10% ammonium sulphamate (0·15 ml) to cold 0·005 *M* *p*-nitroaniline (10 ml)]. The colour was read at 520 mμ. This method was successfully applied to the determination of **I** in blood, urine and brain. CHEM. ABSTR.

3436. **Isolation, characterisation and quantitative determination of organic bases (pharmaceuticals) with the aid of disulphimides [disulphonamides].** F. Runge, H.-J. Engelbrecht and H. Franke (Inst. f. Tech. Chem., Univ. Halle, Germany). *Pharmazie*, 1957, **12** (1), 8–13.—Disulphonamide salts, such as $(p\text{-Cl}\cdot C_6H_4\cdot SO_2)_2N^-$ and $(p\text{-}NH_2\cdot C_6H_4\cdot SO_2)_2N^-$ can be used for the characterisation of many pharmaceutical bases. The m.p. of 35 such derivatives are given, together with directions for the prep. of the disulphonamides. In many cases, the method can be used for the quant. gravimetric determination of the bases. A. R. ROGERS

See also Abstracts 3221, 3398, 3399, 3400, 3464.

Food

3437. **Determination of traces of zinc in foodstuffs.** A. C. Francis and A. J. Pilgrim (Rowntree & Co. Ltd., York, England). *Analyst*, 1957, **82**, 289–292.—The sample (5 g) is ashed with 0·5 ml of conc. H_2SO_4 at a low temp. (550°), the ash is dissolved in 2·5 ml of 5 *N* HCl, 5 *N* ammonium acetate (8 ml) is added and the soln. is diluted to 100 ml. A blank soln. is prepared similarly with omission of the sample. By a method described, an approx. estimation of the content of Zn of the test soln. is made. A vol. ≡ 20 to 100 μg of Zn is taken and the same vol. of a blank soln. Each soln. is mixed well with 10% $Na_2S_2O_3$ soln. (1 ml). A vol. of a standard soln. of Zn containing the amount estimated to be present in the sample soln. is added to the blank soln. and the same vol. of water to the sample soln. The two soln. are then shaken with the same vol. of a chloroform soln. of dithizone for exactly 3 min. The colours of the two extracts

are compared and a standard soln. of Zn is added to one or the other until exact matching is achieved.
A. O. JONES

3438. **Use of an anion-exchange resin in the determination of traces of lead in food.** E. I. Johnson and R. D. A. Polhill (Dept. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1957, **82**, 238–241.—An aliquot of a soln. of the ash in 2 *N* HCl (equiv. to >40 μg of Pb) is applied to a column of the chloride form of Amberlite IRA-400 and followed with 25 ml of *N* HCl. The column is eluted with 0·01 *N* HCl. The Pb content of the eluate is determined by extraction with a chloroform soln. of dithizone, and measurement of the extinction at 520 mμ. Comparison of the results with those obtained by other methods indicates satisfactory accuracy. A. O. JONES

3439. **Simultaneous measurement of glucose and fructose.** W. L. Brown, M. K. Young and L. G. Seraile (Brooke Army Med. Center, Fort Sam Houston, Tex., U.S.A.). *J. Lab. Clin. Med.*, 1957, **49** (4), 630–634.—The modification of the anthrone method described depends on the different rates of reaction of anthrone with fructose and glucose at 75° and at 100°; the concentrations of the two sugars are calculated by solving simultaneous equations based on the optical densities produced under standard conditions. The method is suitable for biological samples and may be used in the study of intermediary carbohydrate metabolism.
W. H. C. SHAW

3440. **Polarography of pectins.** A. L. Markman and A. S. Gorokhovskaya (Central Asiatic Polytech. Inst.). *Zavod. Lab.*, 1957, **23** (3), 289–295.—The method is based on measurements of the degree of suppression of the maximum of the polarographic wave of O. In pure pectin soln. the concn. ($<1\%$) of pectin $\propto \delta^{\frac{3}{2}}$, where δ is the depression of the wave. In complex mixtures separations are made by extractions with hot water, 96% ethanol, ether, etc. G. S. SMITH

3441. **Electrochemical method for the determination of lipoxidase activity in cereals and cereal products.** M. Rohrlich, F. Tödt and G. Ziehmann (Versuchsanstalt f. Getreideverwertung, Berlin). *Fette, Seif., Anstrichmitt.*, 1956, **58** (12), 1057–1059.—An aliquot of an aq. extract of the material to be tested is added to an emulsion of linoleic - linolenic acid, saturated with atmospheric oxygen and contained in a specially constructed electrical cell which generates a current proportional to the oxygen content of the electrolyte. As the acid is oxidised, the dissolved oxygen in the cell is used up and the current decreases. The rate of oxidation can thus be measured. The cell consists of a vessel of about 90 ml capacity with a gold-amalgam cathode and an iron anode; the anode compartment is filled with a saturated soln. of KCl and is separated from the cathode compartment, containing the test soln., by an earthenware diaphragm. Measurements of the lipoxidase activity of various cereal meals agree with values recorded in the literature.
E. HAYES

3442. **The proximate analysis of wheat flour carbohydrates. I. Methods and scheme of analysis.** J. R. Fraser, M. Brandon-Bravo and D. C. Holmes (Dept. Gov. Chem., Clement's Inn Passage, London). *J. Sci. Food Agric.*, 1956, **7** (9), 577–589.—Procedures are recommended for the determination of starch, sugar, hemicellulosic material and cellulosic material. Starch is determined by polarisation and pentosans by the orcinol - Fe colour reaction. An enzymolytic and a modified fibre method are suggested for cellulose. The determination of the "total reducing value" after hydrolysis with $CaCl_2$ soln. affords a check on the results.

II. The analysis of the carbohydrate fractions of different flour types. J. R. Fraser and D. C. Holmes. *Ibid.*, 1956, **7** (9), 589–594.—Representative samples of flour of different extraction rate have been examined by the methods proposed in Part I.
G. B. THACKRAY

3443. **Flour testing. II. An alternative method for using the Brabender Farinograph and Extensograph for testing bread flours.** R. Bennett and J. B. M. Coppock (British Baking Ind. Res. Ass., Chorleywood, Herts., England). *J. Sci. Food Agric.*, 1956, **7** (12), 764–767.—A new technique is described for using the Brabender Farinograph and Extensograph, involving (*i*) the inclusion of yeast in the dough, (*ii*) the measurement of water absorption after a fermentation period, (*iii*) moulding the dough after fermentation and before stretching. This yields results of greater practical value in respect of bread flours with varying degrees of treatment. G. B. THACKRAY

3444. **Determination of total and inorganic bromide residues in fumigated products.** D. A. Mapes and S. A. Shrader (Dow Chemical Co., Midland, Mich., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 189–191.—The sample that has been treated with ethylene dibromide or methyl bromide is refluxed with ethanol and ethanolamine to hydrolyse the halides, and the ethanol is removed by evaporation in the presence of NaOH. The residue is then heated at 600° with NaOH and Na_2O_2. Bromide extracted from the ash is determined by the method of van der Meulen (*Chem. Weekbl.*, 1931, **28**, 82) and Kolthoff and Yutzy (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 75). Inorganic bromide in materials fumigated with methyl bromide or ethylene dibromide is determined by the method of Shrader *et al.* (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 1). Recoveries of 78 to 90% of added ethylene dibromide are reported.
A. A. ELDRIDGE

3445. **Total volatile halide determination of aggregate residue of carbon tetrachloride, ethylene dichloride and ethylene dibromide in fumigated cereal products: determination of bromide by the Kolthoff - Yutzy - van der Meulen procedure and of ethylene dichloride by difference. I. Combustion procedure.** D. A. Mapes and S. A. Shrader (Dow Chemical Co., Midland, Mich., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 180–185.—The organic halides are removed from a mixture of grain, water, H_2SO_4 and tungstophosphoric acid by means of a current of O, which is then passed over platinum gauze at 900°, the halogens being absorbed in a soln. of Na_2CO_3 and Na formate. Total Cl^- plus Br^- is then determined by the micro-Volhard method and Br^- by the procedure of Kolthoff and Yutzy (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 75); Cl^- is found by difference. Recoveries reported are—$C_2H_4Cl_2$, 73 to 110%; $C_2H_4Br_2$, 72 to 92%.

II. Ethanolamine - sodium reduction procedure. H. W. Conroy, V. E. Munsey and L. L. Ramsey (Food and Drug Admin., Kansas City, Mo., U.S.A.). *Ibid.*, 1957, **40** (1), 185–189.—In Ramsey's procedure for isolating the CCl_4, ethylene dichloride and

ethylene dibromide (*Anal. Abstr.*, 1957, **4**, 3446), the acetone is replaced as absorbent by a mixture of dioxan and ethanolamine (5:1). The halide soln. is refluxed, at first alone and then with Na (*cf.* Winteringham, *J. Soc. Chem. Ind.*, 1942, **61**, 186), and the resulting inorganic halide is determined by Volhard titration. Recoveries of ethylene dibromide added to wheat suspensions were 87 and 88% and to flour suspensions 82 and 86%. A. A. ELDRIDGE

3446. **Colorimetric determination of carbon tetrachloride in fumigated cereal products.** L. L. Ramsey (Food and Drug Admin., Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 175–180.—Residual CCl_4 on grain is determined by passing air through a boiling acidified mixture of grain and water containing tungstophosphoric acid, removing excessive entrained water vapour in a condenser at 55° to 65°, and absorbing the CCl_4 from the air in acetone at 0°. The CCl_4 is then determined colorimetrically with pyridine (*cf.* Rogers and Kay, *J. Ind. Hyg. & Toxic.*, 1947, **29**, 229). The recovery of CCl_4 was essentially quantitative. A. A. ELDRIDGE

3447. **Determination of carbon disulphide in fumigated cereal products. I. Adaptation of Lowen's dithiocarbamate method.** C. L. Dunning (Diamond Alkali Co., Painesville, Ohio, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 168–171.—Residues of CS_2 remaining after treatment of grain with CCl_4 and CS_2 (80:20) are determined by digesting the sample with HCl (1:1) and aspirating the CS_2 through a Pb acetate (10% w/v) trap into a soln. of Cu acetate (0·05 g) in ethanol (1 litre) containing diethylamine (1 ml) and triethanolamine (20 ml). Quantities larger than 25 p.p.m. are collected at −7° in *iso*propyl alcohol to which a similar reagent (containing also water and *iso*propyl alcohol) is added. The extinction of the resulting soln. is determined at 425 mμ with reference to a standard curve. Recoveries of 87·6 to 92·5% of added CS_2 are reported.

II. A second adaptation of Lowen's dithiocarbamate method. G. E. Keppel and V. E. Munsey (Food and Drug Admin., Minneapolis, Minn., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 171–174.—Minor modifications in procedure led to recoveries of 86 to 103% of added CS_2. The technique is described and apparatus is illustrated, with dimensions. A. A. ELDRIDGE

3448. **The agar diffusion precipitin test as a means of recording evidence of meat adulteration.** R. M. Pike and S. E. Sulkin (Univ. Texas Med. Sch., Dallas, U.S.A.). *J. Lab. Clin. Med.*, 1957, **49** (4), 657–660.—The tube dilution method with rabbit anti-horse serum for the detection of horse meat is adapted as an agar plate diffusion test. The resulting plates can be photographed for use in medico-legal proceedings. The procedure will detect 5% of horse meat added to beef. W. H. C. SHAW

3449. **Displacement separation of some of the component fatty acids of milk fat.** S. Kuramoto, J. J. Jezeski and R. T. Holman (Minnesota Univ., St. Paul, U.S.A.). *J. Dairy Sci.*, 1957, **40** (3), 314–322.—The separation of nine fatty acids from milk fat by carrier displacement chromatography is described. After saponification of the sample, an aliquot of the soap solution is placed on a charcoal - Hyflo-Supercel column, the fatty acids are liberated with acid, and the column is then developed with a solution of a suitable displacer. A modification of the Tiselius - Claesson apparatus is used and the fatty acids are detected by interferometric methods. W. H. C. SHAW

3450. **Determination of water content in butter with and without distributing agents.** A. Seuss, F. Kiermeier and F. J. Brunner (Süddeutschen Versuchs- und ForschAnstalt für Milchwirtschaft, Weihenstephan, Germany). *Z. LebensmittUntersuch.*, 1957, **105** (2), 89–98.—The methods of determination of water in butter have been statistically examined, including the drying-oven method at 103° to 105° with and without NaCl or sand as distributing agent, and the rapid method at 130°, and are compared in relation to accuracy and time expenditure. The results in which NaCl (of various grades) were used were significantly lower than when sand was used and the drying time was longer. S.C.I. ABSTR.

3451. **Application of the thiobarbituric acid method for the determination of oxidative changes in butter.** E. Černá, J. Mašek and M. Vedlich (ForschAnstalt für Milch und Eier, Prague). *Z. LebensmittUntersuch.*, 1957, **105** (2), 86–88.—Best results were obtained with a modification of the Patton - Kurtze method. The fat (3 g), after being melted at 45° and filtered, is shaken thoroughly with water (8 ml), thiobarbituric acid reagent (6 ml) and trichloroacetic acid (3 ml) in a test-tube, placed in a boiling-water bath for 10 min., then rapidly cooled. The colour is measured in a colorimeter at 530 mμ (green filter) or by comparison with a prepared series of cobalt nitrate solutions. S.C.I. ABSTR.

3452. **Detection of refining of lard. III. The ultra-violet absorption spectrum.** H. P. Kaufmann, J. G. Thieme and F. Volbert (Dtsch. Inst. f. Fettforschung, Münster, Westphalia, Germany). *Fette, Seif., Anstrichmitt.*, 1956, **58** (12), 1046–1057.—The method of detecting lard that has been refined with bleaching earth, by measuring the absorption spectrum in hexane over the range 220 to 300 mμ, is further studied (*cf. Anal. Abstr.*, 1957, **4**, 3122, 3123). Samples of genuine lard obtained from pigs, fed with widely different diets, all exhibited "Q" and "T" values well below those found for refined inferior lard. The method is not invalidated by oxidation of the lard, but "white grease" loses the characteristic triene structure when it is highly oxidised by aeration. This characteristic structure is not produced by alkali refining or by deodorisation, but only by bleaching earth. The effect of the bleaching earth is most marked with inferior oxidised lard. The method cannot be used to determine the amount of refined inferior lard in mixtures with genuine lard, but when the content of refined lard is between 10 and 20%, the "T" and "Q" values obtained indicate refined inferior lard. E. HAYES

3453. **A modified method for the quantitative estimation of diphenyl in citrus fruits.** W. L. Stanley, S. H. Vannier and B. Gentili (Fruit and Veg. Chem. Lab., Agric. Res. Service, Pasadena, Calif., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 282–286.—Improved methods of solvent purification, sample preparation, volume dilution, and filtration have been devised and incorporated in the chromatostrip - spectral procedure (*cf.* Kirchner *et al.*, *Anal. Abstr.*, 1955, **2**, 767) for determining diphenyl. A. A. ELDRIDGE

3454. **The pectic substance in tea. I. Determination of pectin in tea leaves.** Toshio Nakabayashi and Hiroatsu Washiyama (Fac. Agric., Shizuoka Univ., Iwata, Shizuoka Prefecture). *J. Agric. Chem. Soc. Japan*, 1956, **30** (7), 401–405.—The colloidal titration of pectin was studied with methylglycol chitosan (**I**) and K poly(vinyl alcohol) sulphate (**II**). Pectin in ammonium oxalate is catalytically decomposed with invertase (**III**) at pH 3·8; this action is suppressed by the addition of a small amount of NaOH. Neither tannin nor protein is decomposed by **III**. The results agree well with those obtained by the Ca pectate method, and the time taken for estimation is reduced to one third. *Procedure*—Tea leaf (1 g) is washed with methanol and extracted with 0·5% aq. ammonium oxalate soln. (2 × 100 ml) for 3 hr. and a 5-ml portion of the extract is neutralised with 0·5 *N* NaOH to phenolphthalein. The product is treated with *M*/15 acetate buffer soln. (pH 3·8) (1 ml) and digested with **III** soln. (1 + 100, 1 ml) for 1 hr. at room temp.; it is then mixed with 0·5 *N* NaOH to produce a 0·1 *N* soln., set aside for 1 hr. and neutralised with 0·5 *N* acetic acid. This soln. is treated with 0·0005 *N* **I** soln. (5 ml) and a few drops of toluidine blue soln. (0·2%) and titrated with 0·0025 *N* **II** until the colour changes from blue to purple. The blank test is carried out by making the soln. basic immediately after the addition of **III**. For the determination of sol. pectin, the leaf is extracted three times with water (100, 50 and 50 ml, for 2, 1 and 1 hr., respectively), and a 20-ml portion is treated as described above. K. Saito

3455. **Rapid determination of carbon dioxide in lager-tank beer.** S. Rischbieter (Wetzlar/Lahn, Germany). *Brauwelt, B*, 1957, **97** (29–30), 459–461.—The apparatus designed for this determination comprises a glass cylinder, closed above and below with brass caps, each fitted with a tap. A pressure gauge is mounted on the upper cap, and a thermometer is fitted inside the cylinder. After the air in the cylinder has been replaced by CO_2, and a pressure (greater than the pressure in the lager-tank) of CO_2 has been established in the apparatus, the orifice of the lower tap is dipped below the surface of the beer, the pressure is released through this tap, and sufficient of the sample to occupy $\simeq$96% of the capacity of the cylinder is drawn in by slow suction through the upper tap. After closing both taps and gently agitating the apparatus, the pressure over the beer, and the temp. of the beer are read and converted into percentage (w/w) of CO_2 by reference to a table which is attached to the cylinder. The results obtained by the procedure agree satisfactorily with those of chemical determinations. P. S. Arup

3456. **New methods of analysis [of brewing materials] and their evaluation.** A. Schmal (Versuchsstation Schweiz. Brauereien, Zurich, Switzerland). *Schweiz. Brau. Rundschau*, 1956, **67** (3), 35–37.—This is a review of certain methods of analysis applied to malt and hops. W. J. Wright

3457. **Gradient elution analysis of hop bitter substances.** L. O. Spetsig and M. Steninger (Forskningslab. LKB, Appelviken, Sweden). *Acta Chem. Scand.*, 1957, **11** (1), 198–199.—Separation of humulones and lupulones by chromatography (*cf. Anal. Abstr.*, 1957, **4**, 1683) can be effected in a single operation by gradient elution. The concn. gradient of the eluent was accomplished by letting 90% (w/w) methanol flow into a constant-vol. mixing chamber loaded with 55% (w/w) methanol, the effluent being fed to the column. The resolution was satisfactory, but less complete than with normal elution. Lupulone and adlupulone were not separated. To overcome errors in the collection of eluate fractions from a chromatographic column and in the determination of the u.v. absorption of each fraction, the column eluate was passed through a 5-mm absorption cell in a Hilger Uvispek spectrophotometer and the extinction at 280 or 325 mμ was recorded automatically by a Brown recorder. W. J. Wright

3458. **Separation and identification of aromatic materials in wine distillation. II. Separation and identification of the higher alcohols.** A. Frey and D. Wegener (Inst. für Landwirtschaft. Technol., Weihenstephan, Germany). *Z. LebensmittUntersuch.*, 1957, **105** (2), 98–104.—The higher alcohols of wine distillates, after oxidation to the corresponding fatty acids, were separated by paper chromatography. The esterified acids of the early fractions are chiefly bound to ethanol. The middle distillate contains large quantities of higher alcohols in free form. Esters of higher alcohols occur in greater ranges in the first half of the middle fractions. Ethyl esters of higher fatty acids appear especially in the later fractions. S.C.I. Abstr.

3459. **Determination of alkalinity of ash of wines and fruit juices.** J. Schneyder (Landw.-Chem. Bundesversuchsanstalt, Vienna). *Mitt. Wein u. Obstbau*, 1957, **7** (1), 26–30.—In comparison with other current methods, the method of Rancke-Madsen and Kjaergard as modified by the author (*Ibid.*, 1956, **6**, 309) gives the most accurate results for the "true alkalinity" of the ash, comprising the alkalinity due to the oxides, carbonates and silicates. P. S. Arup

3460. **Determination of distinctive organic materials added to whisky as markers. VII. Spectrophotometric determination of carbamide [urea].** M. J. Pro, R. A. Nelson and A. P. Mathers (Alcohol and Tobacco Tax Div. Lab., Internal Rev. Service, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 309–315.—The sample and a whisky (10 ml each) containing a known amount of urea are evaporated to 3 ml, diluted, and centrifuged, and an aliquot (5 ml) is treated with xanthhydrol reagent (6 ml). After 3·5 hr. the mixture is centrifuged and the ppt. is collected on a Gooch crucible and dissolved in H_2SO_4 (1:1). After centrifuging the solution its extinction is read at 430 mμ. Recoveries (85 to 114%) of added urea averaged 99·2%. A. A. Eldridge

3461. **Application of gas - liquid partition chromatography to the study of volatile flavour compounds.** W. G. Jennings (California Univ., Davis, U.S.A.). *J. Dairy Sci.*, 1957, **40** (3), 271–279.—The use of a gas - liquid partition column containing polyoxyethylene polypropylene glycol ether supported on prepared Celite is applied to an examination of the volatile components from 10 μl of an ether extract prepared from a commercial starter distillate. The column is maintained at 125° to 130° with He as carrier gas, and the components issuing from the column are detected with a thermal conductivity cell. Ethanol, diacetyl, acetic acid and methyl acetate were identified, and their identity was confirmed by the preparation of derivatives. The limitations of the method are discussed. W. H. C. Shaw

3462. **Lorentz - Lorenz expression as a new analytical constant for fats (ghee) and oils. II. Ghee [butter fat].** A. C. Chatterji and U. Chandra (Chem. Dept., Lucknow Univ., India). *Z. anal. Chem.*, 1957, **156** (1), 1–7.—The use of molecular refraction for detecting adulteration in mustard oil (*cf. Anal. Abstr.*, 1957, **4**, 1685) has been extended to the examination of ghee (butter fat).

M. F. C. LADD

3463. **Analysis of fats from chocolates and chocolate products.** F. T. van Voorst (Keuringsdienst voor Waren, Alkmaar, Holland). *Chem. Weekbl.*, 1957, **53** (15), 170–174.—The following requirements are stated to be essential for judging the quality of the fat isolated from chocolate products—an efficient method of extraction; reliable methods of analysis; and knowledge of the constants of cocoa fat, butter fat and other fats. For Soxhlet extraction, light petroleum (boiling range 40° to 60°), which extracts all fats as well as lecithin, is recommended. To remove the lecithin, the fat should be further extracted with boiling acetone, filtered and dried at 100° to 105°. The usual analytical methods, such as the determination of the iodine and acid values, are described, and tables of constants for cocoa fat, palm-kernel and arachis oils, as well as for soya lecithin are given. P. HAAS

3464. **Colorimetric determination of folic acid in the presence of large amounts of ferrous salts.** E. Häberli, E. Béguin and G. Schenk (Anal. Lab. der Siegfried A.-G., Zofingen, Switzerland). *Z. anal. Chem.*, 1957, **155** (6), 415–417.—Large amounts of Fe^{II} interfere with the U.S.P. method for the determination of folic acid which involves reduction, diazotisation and coupling with N-1-naphthylethylenediamine. By carrying out the reaction in the presence of 3% of H_3PO_4, as much as a 500-fold excess of Fe^{II} can be masked as the stable complex phosphate. A. R. ROGERS

3465. **Determination of ascorbic acid in foodstuffs by paper chromatography.** G. Cerutti (Ital. Chemotherap. Res. Inst., Milan, Italy). *Chim. e Ind.*, 1957, **39** (1), 14–16.—Ascorbic acid, *e.g.*, in wines, beers or syrups, is separated by paper chromatography (ascending), with *n*-butanol as solvent, and the band corresponding to the R_F value of the acid (0·30 to 0·40) is titrated after stabilisation with oxalic acid. Determination from the area of the spots, revealed by treatment with dichlorophenolindophenol, or by long exposure to air, is less accurate. L. A. O'NEILL

3466. **Determination of true vitamin B_{12} by means of bioautography.** T. P. Verkhovtseva and E. I. Surikova (All-Union Sci. Res. Inst. of Antibiotics, Moscow). *Lab. Delo*, 1957, **3** (2), 24–26.—The soln. to be examined for vitamin B_{12} are placed on paper strips 0·5 cm wide and 25 cm long which are held vertically in a sealed chamber containing a 2 to 3-cm layer of a soln. obtained by mixing 100 ml of *sec.*-butyl alcohol, 100 ml of water, 0·8 ml of conc. aq. NH_3 and 0·5 ml of 5% NaCN soln. Each test soln. is examined in two concn., the ratio being 1 : 2 and the greater concn. being $\geqslant$ 0·2 μg per ml. Comparison is made with a standard soln. containing 0·1 μg per ml. An amount (0·005 ml) of each of these soln. is placed on five parallel paper strips at a distance of 3 cm from the lower end. The ends are fixed 0·5 cm below the surface of the solvent which has already been in the chamber for $\not<$ 3 hr. The chamber is tightly closed and kept at 37° for 20 to 30 hr. The paper strips are then dried for 1 hr. in air and mounted on specially prepared agar plates, containing a sterile culture medium prepared as follows. A mixture of 3 g of NaCl, 2 g of NH_4Cl, 3 g of lactose, 15 g of agar and 1 litre of water (pH 7) is melted on a water bath, then cooled to 48° to 50° and inoculated with *Escherichia coli* 113–3 previously grown for 18 hr. on peptone - agar. The inoculated medium ($\simeq$ 300 ml) is poured on to a sterile glass tray (50 cm $\times$ 30 cm) with a wooden frame. The tray together with the paper strips is kept at 37° for 17 to 20 hr. The diameter of the zone of growth of *Esch. coli* is then measured. A correction is applied if the value for the standard differs from the tabulated value of 17 mm. The content of vitamin B_{12} in the test soln. is calculated from tables. It is essential that the strip width and the vol. of soln. applied be kept constant in all experiments. The errors lie between $\pm$ 5 and 15%.

G. S. SMITH

3467. **Determination of α-tocopherol by chromatography on secondary magnesium phosphate.** F. Bro-Rasmussen and W. Hjarde (Statens Vitamin Lab., Copenhagen, Denmark). *Acta Chem. Scand.*, 1957, **11** (1), 34–43 (in English).—A quant. chromatographic method is described in which both carotene and vitamin A, as well as other tocopherols, are removed in one step without the need for prior reduction of sterols present; 98 to 100% of α-tocopherol is recovered. The sample is saponified with alcoholic KOH to which pyrogallol has been added to prevent oxidation of the tocopherols. The saponified soln. is repeatedly extracted with ether, the extracts are washed with water, filtered through anhyd. Na_2SO_4, and evaporated without heat in a current of CO_2. The residue is dissolved in light petroleum. From the soln., the α-tocopherol is isolated chromatographically on $MgHPO_4$ and finally determined colorimetrically with $FeCl_3$ and 2 : 2′-dipyridyl in a spectrophotometer at 520 mμ exactly 2 min. after addition of the reagents.

W. J. WRIGHT

3468. **Quantitative determination of the individual tocopherols by chromatography on secondary magnesium phosphate.** F. Bro-Rasmussen and W. Hjarde (Statens Vitamin Lab., Copenhagen, Denmark). *Acta Chem. Scand.*, 1957, **11** (1), 44–52 (in English).—A method is described in which, by chromatographic separation on $MgHPO_4$, ζ-tocopherol (5 : 7-dimethyltocol) is found with α-tocopherol, and ε-tocopherol (5-methyltocol), is estimated with β- and γ-tocopherol. The method is based on that used for determining α-tocopherol (*cf. Anal. Abstr.*, 1957, **4**, 3467) except that chromatography of the unsaponifiable sample is continued beyond elution with 2% ether in light petroleum, gradually increasing concn. of 2% to 7% being used. Successive elution of the individual tocophenols occurs in the order: α-, β- + γ-, and δ-tocopherol. Carotene present is not adsorbed, but passes through the column with the light petroleum. Fractionation curves are shown for various mixtures of synthetic tocopherols, and of natural products and synthetic tocopherols. W. J. WRIGHT

See also Abstracts **3314**, **3253**.

Sanitation

3469. **New method for the determination of carbon monoxide in air.** G. Ciuhandu (Inst. Hyg., Timişoara, Romania). *Acad. Rep. Populare Romîne, Baza Cercetări Ştiinţ. Timişoara, Studii Cercetări ştiinţ., Ser. I*, 1955, **2**, 133–142.—The method is based on the formation of a complex which contains Ag and *p*-sulphamoylbenzoic acid (**I**). The air to be analysed is bubbled through 20% KOH soln. to remove H_2S, then it is passed through a soln. prepared from 0·125 *M* $AgNO_3$ (1 ml), 0·1 *M* disodium salt of **I** (1 ml) and *M* NaOH soln. (0·5 ml) until an incipient yellow colour is observed. The concn. of CO is established by a reference curve. The reagent is not influenced by any other gas except H_2S. CHEM. ABSTR.

3470. **Estimation of toxic gases in air. VII. Chlorine determination.** Tomitaro Fukuyama, Tokuro Sato and Aiko Watanabe. *Bull. Inst. Public Health, Tokyo*, 1955, **4** (4), 10–13.—A simplified method has been devised for the determination of Cl in air with *o*-tolidine (**I**). Mix 1 ml of 0·1% **I** soln. in 1·75 *N* HCl with 20 ml of H_2O, bubble the air through this at 200 to 500 ml per min., incubate at 25° for 5 min. and determine the extinction at 405 mμ or compare the colour intensity with a $K_2Cr_2O_7$ - K_2CrO_4 soln. in phosphate buffer at pH 6·45. The colour is stable for 25 min. at 20° to 25° in the presence of 0·075 to 0·2 *N* HCl. This method is applicable for up to 20 p.p.m. of Cl.

VIII. Acetone. Tomitaro Fukuyama, Tokuro Sato and Aiko Watanabe. *Ibid.*, 1956, **5** (1), 2–7.—An improved salicylaldehyde (**II**) method was devised. Pass the air through 20 ml of 1% $NaHSO_3$ at a rate of 100 ml per min. (99·8% is dissolved). Mix a 5-ml portion with 5 ml of dil. KOH soln. and warm at 30° for 10 min. Add 1 ml of **II**, heat in a boiling-water bath for 15 min., cool immediately, dilute to 15 ml with H_2O and determine the intensity of colour 15 to 20 min. after the dilution.

IX. Formaldehyde. Tomitaro Fukuyama, Tokuro Sato, Aiko Watanabe and Hideko Kimura. *Ibid.*, 1956, **5** (3), 1–6.—The Schryver hydrazone method (*cf.* Jacobs, "Analytical Chemistry of Industrial Poisons, Hazards and Solvents," 1949) for determining formaldehyde is improved by replacing HCl with 15 *N* H_2SO_4. It was found advantageous to carry out the colorimetric procedure at 20° to 25°. CHEM. ABSTR.

3471. **Determination of lead, zinc and thallium in air by a spectrographic method.** M. S. Bŭkhovskaya and M. D. Babina. *Gigiena i Sanit.*, 1956, (7), 26–30; *Ref. Zhur., Khim.*, 1957, Abstr. No. 4749.—The three-standards method was used. On excitation of the spectrum in an a.c. or d.c. carbon arc, the sensitivity of the method is 0·01 mg of Pb in the arc or 0·03 mg in the sample, and 0·01 and 0·03 mg of Zn and 0·003 and 0·01 mg of Tl, respectively. On excitation of the spectrum in a condensed spark the sensitivity is considerably lower. The determination of Pb is interfered with by Sn, Cu and Zn; the sensitivity is no higher than that of the nephelometric determination as $PbCrO_4$. Zinc may be determined in the presence of fivefold quantities of Cu, but the sensitivity is lower than that of the chemical method. The conditions are described for the determination of Pb, Tl (0·01 mg) and Zn. C. D. KOPKIN

3472. **Micro-determination of toluene-di*iso*cyanate in the atmosphere.** K. Marcali (E. I. du Pont de Nemours & Co., Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 552–558.—The method is based on the rapid hydrolysis of the toluene-di*iso*cyanate (**I**) to the corresponding diamine, diazotisation, and coupling of the stable diazo compound with N-1-naphthylethylenediamine. The reddish-blue colour produced is measured at 550 mμ. As little as 0·01 p.p.m. of toluene-2:4-di*iso*cyanate can be detected and a field kit is described which is sensitive to 0·02 p.p.m. A method for the determination of 3:3′-di*iso*cyanato-4:4′-dimethylcarbanilide (**II**) in the presence of **I** is also described. It is based on the formation of a yellow colour when **II** is absorbed in an aq. soln. of 2-ethoxyethanol containing $NaNO_2$ and boric acid. The extinction is measured at 450 mμ, and as little as 0·03 p.p.m. of **II** can be determined. G. P. COOK

3473. **Microphotometer turbidity analysis [of water].** W. R. Conley and R. W. Pitman (General Electric Co., Hanford Atomic Products Operation, Richland, Wash., U.S.A.). *J. Amer. Wat. Wks Ass.*, 1957, **49** (1), 63–67.—A photometer used to measure reflected light was adapted for filter plant control purposes. Basically, the instrument comprised a light source, a photocell and an amplifier. The photocell was set at an angle of 15° from the straight light path, this angle being found to provide max. sensitivity and reproducibility. The scattered light falling on the photocell gave rise to a current which was amplified and then measured in terms of turbidity. The apparatus was standardised with bentonite suspensions of known turbidity values. S.C.I. ABSTR.

3474. **Determination of carbon dioxide in water by conductivity measurement.** C. A. Noll and J. W. Polsky. *Tappi*, 1956, **39** (1), 51–54.—An apparatus is described in which the CO_2 content of a sample is estimated by measurement of the conductivity of a soln. of $Ba(OH)_2$ before and after absorption of the CO_2. The sample containing between 25 and 75 mg of CO_2 is measured into the decomposition flask of the apparatus holding 200 ml of water, which has been first prepared by drawing a stream of dry CO_2-free air through it until the conductivity of $Ba(OH)_2$ soln. in each of two Woulfe bottles arranged in series is constant. After addition of H_2SO_4 soln. (5%) (5 ml), the decomposition flask is heated while a steady flow of air is drawn through the apparatus. The air passes first through a wash-bottle containing conc. H_2SO_4, then over Ascarite to absorb CO_2, and enters the decomposition flask through a trap. The CO_2 liberated from the sample is scrubbed first with chromic acid soln. (50 g in 150 ml of 33% H_2SO_4), then with KI soln. (20%) and is passed into the absorption bottles. The experiment is continued until constant readings for the conductivity of the soln. in the bottles are obtained (about 20 min.). The sum of the changes in conductivity of the soln. is equivalent to the CO_2 absorbed. The method can be adapted for the determination of organic carbon in water by oxidation with chromic acid and measurement of the CO_2 evolved. D. G. FORBES

3475. **Salinity and the biochemical oxygen demand of estuary water.** J. Grindley and A. B. Wheatland. *Wat. Sanit. Engr.*, 1956, **6**, 10–14.—Erratic results obtained when determining the 5-day B.O.D. of samples of estuary water led to an investigation into the effect of salinity on the B.O.D. The B.O.D. of samples was determined by the 5-day test and over a longer period in respirometers under

varying conditions of salinity and available nutrients, and the degree to which nitrification had progressed at the end of the experiment was determined. The limitations of the 5-day B.O.D. test for saline water samples and the extent to which it is affected by the degree of nitrification are discussed. It is concluded that pollution of saline water can be determined more accurately by measuring the B.O.D. in a respirometer over a much longer period, or by determining the concn. of carbon and nitrogen compounds. WATER POLLUTION ABSTR.

3476. **The determination of the oxygen content of boiler feed water.** C. Janssen and G. B. Smit (N.V. tot Keuring van Electrotech. Materialen, Arnhem, Netherlands). *Anal. Chim. Acta*, 1957, **16** (3), 276–284.—Available methods are briefly reviewed and some results are compared. The authors have adopted the U.S. Navy method (Adams *et al.*, *Brit. Abstr. C*, 1945, 10) for the treatment of samples and the dead-stop end-point method of titration (Foulk and Bawden, *J. Amer. Chem. Soc.*, 1926, **48**, 2045). Trace quantities of Cu^+, Cu^{2+}, NO_3^-, hydrazine and SO_3^{2-} do not interfere. Interference due to Fe and NO_2^- can be eliminated by treating the sample with ion-exchange resins. W. C. JOHNSON

3477. **Determination of nitrates in boiler water by 1:3-xylen-4-ol(2:4-xylenol).** J. S. Swain (Power Station, Newton Abbot, S. Devon). *Chem. & Ind.*, 1957, (16), 479–480.—The sample is mixed with 85% H_2SO_4 and 2:4-xylenol. The xylenol becomes nitrated to 5-nitro-2:4-xylenol, which is steam-distilled into dil. NaOH to give a coloured salt. The degree of coloration is measured absorptiometrically. The depth of colour is affected by temp., which should be maintained at 20°. No interference is caused by the presence of $NaNO_2$ (up to 20 p.p.m.) in the boiler water.

W. J. WRIGHT

3478. **Rapid determination of nitrates in boiler water.** R. L. Babkin and K. P. Epeĭkina. *Energetik*, 1956, (8), 13–14; *Ref. Zhur., Khim.*, 1957, Abstr. No. 4790.—The method is based on the direct titration of NO_3^- by a soln. of indigo carmine in a strongly acid medium. C. D. KOPKIN

3479. **Measurement of small quantities of hydrocarbon in water.** C.-G. Lindgren (Nat. Inst. Publ. Health, Stockholm, Sweden). *J. Amer. Wat. Wks Ass.*, 1957, **49** (1), 55–62.—Petroleum and oil are estimated in water to a level of 0·1 mg per litre with an accuracy of 0·05 mg per litre by extraction with CCl_4, chromatography, and i.r. absorption measurements at 3·38, 3·42 and 3·51 μ, absorption of the water being known. In other cases chromatography with alumina enables estimation to 0·2 mg per litre to be effected with an accuracy of 0·1 mg per litre. S.C.I. ABSTR.

3480. **Volatile acids [in sewage] by direct phosphoric acid distillation.** J. E. Frook (Sewage Treatment Plant, Toledo, Ohio). *Sewage Ind. Wastes*, 1957, **29** (1), 18–23.—The sample of sludge is freed from solids by settling and decanting, followed by flocculation with $Al_2(SO_4)_3$ and $Ca(OH)_2$ at pH 7·5 and filtration. The clear filtrate (150 ml) is placed in a Kjeldahl flask with 50 ml of 85% aq. H_3PO_4 and 5 ml of saturated Ag_2SO_4 solution, and distilled into a graduated cylinder until all the volatile acids are in the distillate. The system is purged by distilling 5 to 10 ml of water from a fresh Kjeldahl flask and the total condensate is titrated at $\simeq$100° with 0·1 *N* NaOH to phenolphthalein. Recoveries of 98% of acetic, propionic, *n*-butyric, *n*-valeric, and *n*-hexoic acids are possible. A blank titration is carried out and results are expressed as p.p.m. of acetic acid. S.C.I. ABSTR.

3481. **Colorimetric micro-determination of chloropicrin in water.** R.-M. Fournier and M. Person. *Chim. Anal.*, 1957, **39** (4), 155–158.—Chloropicrin forms deeply coloured compounds when mixed with KCN and phloroglucinol in a pyridine medium. The reagent must be kept in an amber bottle and freshly made each day. The colour is measured by a Rouy photo-electric colorimeter. Waste waters from the manufacture of chloropicrin contain (in addition to chloropicrin), Ca picrate, $CaCl_2$, and $Ca(OH)_2$; only picrate ions cause interference with the colour. Chloropicrin is separated by passing the water through activated carbon and subsequent extraction with ligroin, which is a good solvent for chloropicrin, but not for picrates, is immiscible with water, does not react with the reagent and produces no emulsion with pyridine. A yield of 75% is obtainable from soln. containing 10 to 40 μg of chloropicrin per ml.

W. J. WRIGHT

3482. **Studies on mineral springs. IV. Determination of sodium and potassium in acid springs by ion exchange and flame photometry.** Yasushi Mashiko (Chem. Inst., Hot Spring Research Centre, Kashiwagi, Shinjuku-ku, Tokyo). *J. Pharm. Soc. Japan*, 1956, **76** (11), 1272–1275.—When Na^+ and K^+ (each $\simeq$ 0·2 m*M*), adsorbed on Amberlite IR-120, are eluted with 0·5 *N* HCl, the first 10-ml portion is free from metal ions. The second 50-ml portion contains only Na^+, and the third 100 ml Na^+ and K^+. The last two portions are submitted to flame photometry as described previously (*cf. Anal. Abstr.*, 1957, **4**, 290, 714). Magnesium, Ca, Fe^{3+} and Al^{3+} (each 0·5 milli-equiv.) remain on the resin. This method is applicable to the analysis of hot springs containing H^+ (pH < 2), Fe and Al; the cations are adsorbed on Amberlite IR-120 from 0·1 *N* H_2SO_4 soln. K. SAITO

3483. **Review of the literature of 1956 on sewage, waste treatment and water pollution. I.** Committee on Research, Federation of Sewage and Industrial Wastes Associations. *Sewage Ind. Wastes*, 1957, **29** (5), 497–523.—This review contains a section on analytical methods. N. E.

3484. **Recommended methods for the analysis of trade effluents. Method for the determination of combined nitrogen.** Joint A.B.C.M. - S.A.C. Committee on Methods for the Analysis of Trade Effluents. *Analyst*, 1957, **82**, 276–284.—For ammoniacal N, a distillation method is recommended, followed by nesslerisation or titration. Albuminoid N is determined by distillation with alkaline $KMnO_4$ soln. and subsequent nesslerisation. For the determination of organic N, a preliminary removal of NO_2^- or NO_3^- is effected by reduction to NH_3 with Devarda's alloy and evaporation of the slightly alkaline soln. The organic N is then converted into NH_3 by the Kjeldahl method and, after distillation, is determined by nesslerisation or titration. Total unoxidised N in the absence of NO_2^- and NO_3^- is determined by the Kjeldahl method, with Na_2SO_4 - $CuSO_4$ as catalyst. If NO_2^- and NO_3^- are present, they are reduced to NH_3 by Devarda's alloy, the NH_3 is removed by

boiling and the liquid is then subjected to the Kjeldahl procedure, the amount of ammoniacal N previously found being added to the result. Nitrite N is determined by the Griess - Ilosvay method and nitrate in the residue from the determination of ammoniacal N by reduction to NH_3 and subsequent distillation. A. O. JONES

See also Abstracts 3314, 3348.

Agriculture and Plant Biochemistry

3485. **Method of wet ashing for analyses in agricultural chemistry.** K. Scharrer and H. Munk (Justus Liebig Hochschule, Giessen, Germany). *Agrochimica*, 1956, **1**, 44–55.—Wet ashing with $HClO_4$ shows advantages in speed and accuracy over dry ashing and other wet oxidation procedures for analyses of oxidation-resistant organic materials. For 1 to 5 g of sample with 5 ml of $HClO_4$, 2 to 5 ml of H_2SO_4 and 15 to 40 ml of HNO_3, oxidation is complete in 60 min. With 100 ml of HNO_3 and 10 ml of $HClO_4$ per 5 g of sample, recoveries of S of 98 to 102% were obtained on a variety of organic compounds. Compared with this method, dry ashing of various plant materials at 500° showed losses of K of 0 to 66%; of Zn, 7 to 50%, and Mo, 20 to 76%, due to vaporisation of salts or formation of insoluble complex silicates. E. C. APLING

3486. **The use of nitrilotriacetic acid (Complexone I) in the polarographic determination of trace elements in the ash of plants.** R. Pleticha (Forsch. Inst. f. Pflanzenprodukt., Prague-Ruzyně, Czechoslovakia). *Pharmazie*, 1957, **12** (3), 131–135.—A soln. of nitrilotriacetic acid (0·4 g) in 12·5% aq. NH_3 (10 ml) is used as supporting electrolyte for the polarographic determination of Cu, Zn and Fe present as trace elements in plant ash. The only preliminary treatment required is the removal of silicate. The method of standard additions is used for the calculation of concn. from wave-heights. In suitable cases, Pb, Sb, Tl, Cd and Bi can also be determined. A. R. ROGERS

3487. **Flame-photometric determination of magnesium in plant material. A study of the emission of magnesium in a highly reducing oxygen - acetylene flame.** K. E. Knutson (Forest Res. Inst. of Sweden, Stockholm). *Analyst*, 1957, **82**, 241–254.—The sensitivity obtained with the 285·2-mμ line emitted by the magnesium atom is greater with a highly reducing oxygen - acetylene carburising flame than with a neutral one. Expt. showed that errors due to interference by PO_4^{3-} and SO_4^{2-} can be largely eliminated by the presence of an excess of Ca^{2+}. The study shows that the flame-photometric determination of Mg in plant material can be precise and satisfactorily accurate for 2 to 10 p.p.m. The coeff. of variation of a single determination was $< \pm 1\%$ and the detection limit was 0·06 p.p.m. A. O. JONES

3488. **Improved formaldoxime method for the determination of manganese in plant material.** E. G. Bradfield (Res. Station, Long Ashton, Bristol, England). *Analyst*, 1957, **82**, 254–257.—The dried and ground plant material (1 g) is digested with HNO_3 until free from solid matter and the digest is then fumed with $HClO_4$ until most of that acid is removed. An aliquot (≡ 10 to 50 μg of Mn) of the diluted filtered digest is mixed with a soln. of N-hydroxyethylethylenediaminetriacetic acid (HEEDTA), the free $HClO_4$ present is neutralised with NaOH, and a formaldoxime reagent (prep. described) is added, followed immediately by NaOH. The mixture is heated on a water bath at 65° for 2 hr. its. vol. is adjusted, and its extinction is measured at 450 mμ and referred to a calibration graph. Comparison of results by this method with those obtained by the periodate method indicate satisfactory accuracy. A. O. JONES

3489. **Micro-estimation of the nitrates in plant tissue and soil extracts.** R. P. Murphy (Johnstown Castle Agric. Coll., Wexford, Ireland). *J. Sci. Food Agric.*, 1957, **8** (4), 231–234.—The NO_3^- are reduced to NH_3, which is determined by the Conway micro-diffusion technique. The method compares favourably with the phenoldisulphonic acid method. The time required for the reduction was approx. 60 min., and 0·2 g of Devarda's alloy was used for amounts of NO_3^- in the order of 10 μg per 1 ml of extract. H. B. HEATH

3490. **Determination of stigmasterol in soya sterols by radioactive isotope dilution.** R. A. Donia, A. C. Ott and N. Drake (The Upjohn Co., Kalamazoo, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 464–467.—Stigmasteryl ^{14}C-acetate of known specific radioactivity was added to soya-bean oil sterols and the whole mixture was acetylated and brominated. The sparingly soluble stigmasteryl ^{14}C-acetate tetrabromide was isolated and its activity determined. The stigmasterol concn. is calculated from the ratio of the initial and final specific activities. Recoveries of stigmasterol from standard samples were generally $> 97\%$ and duplicate determinations on natural samples agreed to within 5%. G. P. COOK

3491. **Determination of stigmasterol in soya-bean sterol mixtures by infra-red method.** J. L. Johnson, M. F. Grostic and A. O. Jensen (The Upjohn Co., Kalamazoo, Mich., U.S.A.). *Anal. Chem.*, 1957, **27** (4, Part I), 468–469.—The i.r. absorption of the *trans* double-bond of stigmasterol at 10·3 μ differentiates it from the structurally similar sitosterols and provides the basis for the determination of stigmasterol in soya-bean sterol mixtures. Results were in agreement with those obtained by a radioactive isotope dilution method and the accuracy, when applied to sterol mixtures, is $\simeq \pm 3\%$, based on the total sample. G. P. COOK

3492. **β-Glycosidase of potato. [Determination of β-glucosidase.]** P. Baruah and T. Swain (Low Temp. Res. Sta., Univ. of Cambridge, England). *Biochem. J.*, 1957, **66** (2), 321–323.—A method is described, with salicin as substrate, for the colorimetric determination of β-glucosidase. The amount of saligenin formed is determined spectrophotometrically at 515 mμ by means of its reaction with 4-aminophenazone, and the amount of the enzyme is ascertained from a standard graph. The method will detect 1 μg of saligenin per 10 ml. J. N. ASHLEY

3493. **Biochemical studies on inositol. I. Isolation of *myo*inositol from yeast and its quantitative enzymic estimation.** F. C. Charalampous and P. Abrahams (Dept. of Biochem., Univ. Pennsylvania, Philadelphia, U.S.A.). *J. Biol. Chem.*, 1957, **225** (2), 575–583.—A new, convenient, enzymic, colorimetric micro-method is described for the determination of inositol. Inositol dehydrogenase,

prepared from *Acetobacter suboxydans* 621, when coupled with pig heart diaphorase reduces 2:6-dichlorophenolindophenol anaerobically in the presence of inositol. The disappearance of the blue colour is followed with a Klett colorimeter, a No. 540 filter and specially adapted Thunberg tubes being used. The optimum range of the method is 0·03 to 0·14 μmole of inositol per ml. Duplicate determinations do not vary by more than 5%. Materials to be analysed for total inositol must first be hydrolysed by boiling with 22% HCl. This treatment destroys or modifies most other compounds so that they can be removed on ion-exchange columns. J. N. ASHLEY

3494. **Notes on the 2:4-dinitrophenylhydrazine method for pyrethrum assay.** B. P. Moore (Pest Infestation Lab., Slough, Bucks.). *J. Sci. Food Agric.*, 1956, **7** (12), 740–744.—The formation, isolation, and estimation of the pyrethrin 2:4-dinitrophenylhydrazones has been studied and the fate of a number of non-insecticidal constituents has been elucidated. The determination of total "pyrethrins" involves only small errors, owing to the similarity of molecular weights. A factor of 1·13 is used (not 1·07 as previously reported, *cf. J. Sci. Food Agric.*, 1954, **5**, 500). G. B. THACKRAY

3495. **Separation and identification of chlorinated organic pesticides by paper chromatography. X. The sensitivity of the chromogenic agent for the detection of pesticides in the chromatogram.** L. C. Mitchell (Food and Drug Admin., Dept. of Health, Educ. and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 294–302.—2-Phenoxyethanol (8% v/v) or soya-bean oil (1% v/v) in ethyl ether was used as the stationary phase and 2:2:4-trimethylpentane, aq. methanol, aq. acetone, or aq. pyridine as mobile solvent. The chromogenic agent was $AgNO_3$ (1·7 g) in 2-phenoxyethanol (10 ml) and ethanol (50 ml) diluted to 200 ml with water. The results of sensitivity tests on 40 pesticides are tabulated. On exposure to strong u.v. light the chromogenic agent detects microgram or microlitre quantities of 37 of the pesticides examined. A. A. ELDRIDGE

3496. **Infra-red spectrophotometric determination of DDT and gamma benzene hexachloride [hexachlorocyclohexane] in cotton dusts.** H. C. Austin, jun., F. L. Bonner and E. A. Epps, jun. (Louisiana Agric. Exp. Sta., Baton Rouge, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1957, **40** (1), 286–293.—The method described is based on the baseline density technique (*cf.* Wright, *Ind. Eng. Chem., Anal.*, 1941, **13**, 1). Standard curves are prepared from DDT (**I**) and hexachlorocyclohexane (**II**) in CS_2, with the addition of S and clay to simulate sample conditions. The **I** peak is measured at 9·86 mμ with baseline points at 9·75 and 10·05 mμ. The **II** peak is measured at 14·53 mμ between points at 14·0 and 14·75 mμ. Samples containing approx. 200 mg of **I** and 120 mg of **II** are extracted with CS_2 for the determination. The accuracy of the results is comparable with that attained by the chromatographic method. A. A. ELDRIDGE

3497. **The colorimetric determination of dinitrophenols.** S. Petrashku and E. Grou. *Biol. Zhur. Akad. RNR*, 1956, **1** (1), 263–267; *Ref. Zhur., Khim.*, 1957, Abstr. No. 4844.—The determination of dinitrophenols (**I**) in insecticides and herbicides is based on the conversion of **I** into aminoquinoneimines and the colorimetric determination of these in aq. soln. As a standard, the corresponding dinitroalkylphenol is used. Measurement is carried out by direct comparison on the Duboscq colorimeter, Lange's photo-electric colorimeter with a blue filter, FEK-M or the Pulfrich photometer. Dissolve 1 to 5 g of **I** in 20 ml of ethanol and make up to 250 ml; for water-soluble preparations of **I** use an aq. soln. Reduce 5 ml of soln. with Zn and H_2SO_4 and then oxidise with $K_2Cr_2O_7$. The mean error of the method is ±3%. C. D. KOPKIN

See also Abstracts 3238, 3254.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

3498. **A mechanical shaker.** A. C. Francis (Rowntree & Co. Ltd., York, England). *Analyst*, 1957, **82**, 293.—A mechanical shaker accommodating two conical separating funnels is described and illustrated. The funnels are held in two carriages that slide on vertical steel guides. By means of a motor with cranks and connecting rods a reciprocating motion is communicated to the carriages. One carriage should be at top dead-centre when the other is at bottom dead-centre. A. O. JONES

3499. **Inert-atmosphere dry box.** T. R. P. Gibb, jun. (Tufts Univ., Medford, Mass., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 584–587.—The welded steel box described can be used when analytical manipulations have to be carried out in an inert atmosphere. For a nitrogen atmosphere a high-voltage sodium arc is used as purifier; for helium or argon, free from N, a lithium arc is used. Constructional details are given. The method of use and tests for water and other impurities are described. G. BURGER

3500. **Float siphons for [separation of] non-conducting liquids.** J. Bové (Lab. de Chim. Biol., Inst. Nat. Agronom., Paris, France). *Anal. Chim. Acta*, 1957, **16** (4), 354–356.—Improvements are reported in the construction and operation of the float siphon (vol. 1 to 5 ml) used in the fractional separation of non-electrolytes, *e.g.*, organic solvents, by automatic fraction-collectors (*cf. Anal. Abstr.*, 1955, **2**, 501). The number of failures is reduced to 0·1% when the new device is used for collecting fractions during the chromatography of organic acids on silica columns. W. J. BAKER

3501. **Simple photometer for quantitative evaluation of paper chromatograms in reflected light.** J. Fellegi and L. Sláma (Res. Inst. Cellulose, Bratislava, Czechoslovakia). *Chem. Zvesti*, 1956, **10** (5), 314–320.—The chromatogram is moved over a slot through which it is illuminated, and the intensity of the reflected light is measured by means of two selenium photocells. By the registration of the intensity of the reflected light, curves are obtained on which the heights of the max. are proportional to the log. of the concn. of the substance on the chromatogram. By adjusting the electric circuit, linear data can be obtained. D-Galactose, D-glucose, D-mannose, L-arabinose, D-xylose and L-rhamnose were determined in quantities of 10 to 100 μg. J. PELIKÁN

3502. **Protein chromatography on calcium phosphate columns.** A. Tiselius, S. Hjertén and Ö. Levin (Inst. Biochem., Uppsala, Sweden). *Arch. Biochem. Biophys.*, 1956, **65** (1), 132–155.—Methods for the preparation of stable columns of calcium phosphate (hydroxyapatite) suitable for the chromatography of proteins are given, and the conditions for the specific elution of proteins on such columns are described. Elution is brought about by stepwise or continuous increase in the concn. of the eluent (phosphate buffer of constant pH) or by displacement. Examples of proteins examined include the strongly coloured proteins phycoerythrin and phycocyanin, bovine serum albumin, ovalbumin, lysozyme, haemocyanin and human γ-globulin. Low-mol.-wt. material was found to show little or no adsorption on the columns. The special features of the procedure discussed include the narrow elution ranges and the marked mutual displacement effects shown by many proteins. A description is given of photographic and television methods of observing directly colourless protein zones on the columns, making use of their absorption of short-wave u.v. radiation. W. H. C. SHAW

3503. **Two-dimensional horizontal paper chromatography.** E. Urbani and G. Scollo-Lavizzari (Ist. Zoolog. ed Anatomia, Univ. Camerino, Italy). *Ric. Sci.*, 1957, **27** (2), 455–457.—The apparatus (illustrated) consists of a Pyrex-glass dish (20 cm × 20 cm × 4 cm), and inside, at one end, is placed a small vessel containing the solvent. The apex of a triangular piece of Whatman No. 1 paper dips into the solvent, while the base is in contact with the edge of a sheet of the same paper supported horizontally over the larger vessel, and bearing the sample to be analysed. The solvent rises by capillarity and on reaching the edge of the square sheet diffuses all over it thereby effecting the first separation. When the solvent front reaches to within 2 or 3 mm from the distal edge, the sheet is dried and replaced in a position at right angles to the original direction and treated with the second solvent. After the second separation the sheet is once more dried and sprayed with the appropriate developer. The first and second solvents had the following compositions—*n*-propanol - aq. NH_3 - acetone - water (60:4:30:20) and phenol saturated with water. The entire apparatus was covered with a glass bell-jar. The developer used was a 0·1% soln. of ninhydrin in *n*-butanol saturated with water. As many as 14 amino acids have been separated by this method. P. HAAS

3504. **Cumulative recorder for chromatographic gas analysis.** E. Leibnitz, H. Hrapia and H. G. Könnecke (Inst. für org.-chem. Ind. Minist. für chem. Ind., Leipzig). *BrennstChemie*, 1957, **38** (1), 14–16.—Gases successively displaced from a chromatographic column are swept by a current of CO_2 into a nitrometer connected to a side-tube in which a piston moves so as to accommodate the accumulating gas at const. pressure and keep the aq. KOH at a level fixed by an electrical contact. The contact is connected to the grid of a valve so that, when it is broken, an e.m.f. is applied, at intervals fixed by a rotating cam, to an electromagnet which, by means of a ratchet wheel, draws out the piston in steps of adjustable frequency until the contact is restored. The piston rod carries a pen which records on a rotating paper cylinder the cumulative volume of gas carried into the nitrometer. If the accumulation is rapid, the frequency of the piston steps may be doubled by using two contacts at slightly different levels. An accuracy of 0·1 vol. % is claimed. A chromatogram shows results for H and hydrocarbons up to C_3 adsorbed by silica gel from cracking gas. A. R. PEARSON

3505. **The employment of a R.Rf. titrimeter in conjunction with Blake's R.Rf. chromatograph zone localiser.** G. G. Blake (Univ. of Sydney, Australia). *Anal. Chim. Acta*, 1957, **16** (3), 238–241.—A conductimetric tube (*cf. Analyst*, 1950, **75**, 689) is incorporated in the circuit of the zone localiser (*cf. Anal. Chim. Acta*, 1956, **14**, 329). This provides a means of determining the quantity of solute in each chromatographic spot after the spot has been extracted and the solute has been identified by its R_F value or by chemical means. W. C. JOHNSON

Optical

3506. **Apparatus for the excitation of powders in the direct-current arc.** J. Noar (Thornton Research Centre, Chester, England). *Spectrochim. Acta*, 1957, **9** (2), 157–158.—In the apparatus described a motor-driven mechanism is used to feed the pelleted sample into the arc through a hollow electrode, in order to allow each component in the mixture to vaporise completely and at about the same rate. The apparatus is simple to use and cleaning takes about half an hour (after 50 to 100 exposures). The sensitivity of detection is good, and samples of less than 1 mg have been analysed. Analytical accuracy has been generally good for the elements so far determined, results in the range 0·1% upwards being usually within ± 10% of chemical results with some exceptions (Na, Ca, Si, Mg, V). K. A. PROCTOR

3507. **The design of infra-red spectrometers.** D. H. Roberts (Plessey Co. Ltd., Caswell Res. Lab., Towcester, Northants.). *Chem. & Ind.*, 1957, (16), 482–485.—Advances in optical research and in the development of i.r. spectrophotometers are reviewed and discussed. W. J. WRIGHT

3508. **Plane parallel plate transmission line Stark microwave spectrograph.** S. A. Marshall and J. Weber (U.S. Naval Ordnance Lab., Silver Spring, Md., U.S.A.). *Rev. Sci. Instrum.*, 1957, **28** (2), 134–137.—Details are given of a parallel plane transmission system for high Stark field microwave spectroscopy. The planes comprise optically flat silvered glass plates separated by plane parallel fused quartz spacers. Field-strength inhomogeneities are so reduced that fields of 25 statvolts per cm produce intense sharp lines. G. SKIRROW

3509. **Adaptation of Beckman model B spectrophotometer as spectrofluorimeter.** G. Nadeau and L. P. Joly (Hôpital Saint-Michel Archange, Quebec, Canada). *Anal. Chem.*, 1957, **29** (4, Part I), 583–584.—Modifications of the Beckman model B spectrophotometer are described. With the addition of an RCA IP28 photomultiplier, a solution of 0·25 μg of quinine sulphate per ml in 0·1 *N* H_2SO_4 gave a scale reading of 100 against a blank of the solvent, with max. photomultiplier sensitivity, a slit width of 1·5 mm and a 100-W mercury lamp. K. A. PROCTOR

3510. **Comparison-type nephelometer.** W. F. Wright (Johns Hopkins Univ., Baltimore, Md., U.S.A.). *Rev. Sci. Instrum.*, 1957, **28** (2), 129–134.—A single light source alternately illuminates

the sample and standard cells. One light-integrating sphere and phototube assembly compares the transmitted beams and a second the scattered beams. The apparatus will operate over a range of five density units. Slight modification makes it possible to measure the state of polarisation of the scattered light for both polarised and unpolarised incident light. G. SKIRROW

3511. **A sensitive photo-electric polarimeter.** B. R. Malcolm and A. Elliott (Courtaulds Ltd., Maidenhead, Berks., England). *J. Sci. Instrum.*, 1957, **34** (2), 48–49.—The design and constructional details are given for a photo-electric polarimeter of high sensitivity. G. SKIRROW

See also Abstract 3473.

Thermal

3512. **Electrically heated and thermostatically controlled oil bath.** J. W. Wood (Lab. of Chem. Pharmacology, National Cancer Inst., Bethesda, Md., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 587–588.—The bath comprises a flat-bottomed stainless-steel beaker, an electrically heated immersion coil of stainless-steel tubing, a thermoregulator, pilot light and switch. Beakers of 3- and 9-quart capacity are convenient, with loadings of 0·5 kW and 1 kW. Heaters and switch are connected in series by glass- or asbestos-fibre-covered wire. The advantage over the mantle-heated type of bath is the ease of raising and lowering without losing contact. A temp. of 300° has been reached with a wax bath-filling. Constructional details are given. G. BURGER

3513. **Apparatus for the micro-determination of melting-points.** W. G. Jennings (Univ. of California, Davis, U.S.A.). *J. Chem. Educ.*, 1957, **34** (2), 95.—The apparatus comprises an aluminium block with a circular depression machined in the face and wired with 22-gauge nichrome wire insulated with mica tape. The device is used as a microscope hot-stage, the sample being placed in the depression on a circular coverslip. Melting can conveniently be observed with a polarising microscope. S.C.I. ABSTR.

3514. **Time - temperature cooling curves. Simplified determination of compound purity.** K. L. Nelson (Wayne Univ., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1957, **29** (4, Part I), 512–518.—A new, simple and direct method for determining the time at which freezing would have begun in the absence of supercooling is described; inexpensive apparatus is used. A simple graphical method for estimating heats of fusion is included. K. A. PROCTOR

Electrical

3515. **Improved micro-electrophoresis electrode assembly for use in microbiological investigations.** D. E. E. Loveday and A. M. James (Chem. Dept., Chelsea Poly., London). *J. Sci. Instrum.*, 1957, **34** (3), 97–98.—The electrode liquid (KCl soln.) is separated from the suspension under investigation by a sintered glass disc, which permits the use of higher field strengths than do conventional plugs. G. SKIRROW

3516. **Polarographic analysis. XXV. Current-scanning polarography.** Masayoshi Ishibashi and Taitiro Fujinaga (Dept. of Chem., Fac. Sci., Kyoto Univ., Sakyo-ku, Kyoto). *J. Electrochem. Soc., Japan*, 1956, **24** (8), 375–378.—A current-scanning polarograph was set up by inserting a high resistance (10 or 30 megohms) in series with the electrolytic cell. The wave of oxygen in 0·005 *M* KCl, which yields a distinct max., was compared with this apparatus, the Kolthoff - Lingane type and the Heyrovský - Shikata type. The height of the max. increases in this order, not being detected by the use of the first. The process was also examined with a cathode-ray oscillograph, with reference to the relation between current, potential and growth of the mercury drop. It was found that the new apparatus gives perfect control of current electrolysis, in contrast to Heyrovský's type, Kolthoff's being intermediate between the two.

XXVI. Current - potential diagram in the current-scanning polarograph. Masayoshi Ishibashi and Taitiro Fujinaga. *Ibid.*, 1956, **24** (11), 525–528.—The polarogram obtained with the new apparatus differs from those obtained with the others, the shape being markedly affected by foreign ions in the supporting soln. The difference is discussed theoretically. It was found that a recorder of quick response should be used without damping, and the wave height calculated from the upper edge of the wave corresponding to the current at the max. drop surface. K. SAITO

3517. **Polarography with mercury-drop control. II. Application to alternating-current polarography.** Yoshikazu Yasumori (Fac. Agric., Kyoto Univ., Sakyo-ku, Kyoto). *J. Electrochem. Soc. Japan*, 1956, **24** (7), 309–313.—For a.c. polarography of a dil. depolariser, which involves phase difference between condenser current and faradaic current, the use of a synchronised dropping electrode (*cf. Anal. Abstr.*, 1956, **3**, 1946) is suggested. The differential a.c. current is recorded with a Sargent Type Model XXI, with a suitable amplifier and rectifier. A continuous polarogram can be obtained, provided that the natural dropping interval is greater than that when controlled. In the polarography of Pb^{2+} it was found that the constancy of $E_{\frac{1}{4}}$ and the linearity of wave height with concn. are markedly improved by the use of this apparatus. K. SAITO

3518. **Square-wave titrimetry.** L. C. Hall (Univ. of Illinois, Urbana, U.S.A.). *Dissert. Abstr.*, 1956, **16** (9), 1593–1594.—In the procedure described, a square-wave voltage of low-frequency is applied to two similar micro-electrodes in soln. and the slope of the current - voltage curves is measured at zero potential. Theoretical equations are given for the variation in slope when the electrode reaction is reversible, irreversible or controlled partly by electron and mass transfer. Data obtained for the iodine - iodide system are given and discussed in relation to the limitations of the method. The following titrations can be made successfully: (*i*) iodine ($\simeq 5 \times 10^{-6}$ *M*) with arsenite at const. voltage or current, the sensitivity being tenfold greater than for the amperometric method, (*ii*) mixtures of Cl^-, Br^- and I^- ($\simeq 7 \times 10^{-3}$ *M*) with $AgNO_3$ at const. current, the error being within ± 1%, (*iii*) 0·01 *M* KCN with $AgNO_3$ at const. voltage, the error being up to + 0·7%, and (*iv*) $FeSO_4 \cdot (NH_4)_2SO_4$ and $K_4Fe(CN)_6$ in a soln. containing Cl^- and H_2SO_4, the error being + 0·1 to + 1·2% at either const. current or voltage. W. J. BAKER

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	%(v/v)
per cent. (wt. in vol.)	%(w/v)
per cent. (wt. in wt.)	%(w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_{F}
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{3+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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ANALYTICAL ABSTRACTS

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